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Land Treatment of an Oily Waste
Degradation, Immobilization and Bioaccumulation

Cornell Univ., Ithaca, NY

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LAND TREATMENT OF AN OILY WASTE-- DEGRADATION, IMMOBILIZATION AND BIOACCUMULATION

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16. ABSTRACT

An industrial oily waste was applied to field plots in New York to determin loss and immobilization of waste constituents and to determine impact of waste on soi biota. Four replicate plots were established for natural controls, rototilled controls, and low, medium and high application rates. Wastes were applied 06/82, 10/82, and 06/83. In 06/83, plots that had previously received low applications received a very high application. During the study, waste was applied to test plots at seven loading rates that ranged from 0.17-0.5 kg total oil/m^2 or from 0.09 wt%-5.25wt% oil in soil.

Waste application increased soil pH and volatile matter. Half life of total oil in field plots ranged from about 260 to 400 days. Not all of the applied oil was lost. The refractory fraction ranged from 20% to 50% of applied oil. The fraction did not appear to adversely affect soil biota. Napthalenes, alkanes and specific aromatics were lost rapidly, especially in warmer months. The half life generally was less tha 30 days.

Waste applications increased the concentration of several metals in the upper 15 cm of soil. Earthworms bioaccumulated Cd, K, Na and Zn. Accumulation could not be related to waste application and occurred in worms from control plots as well as those receiving waste. Earthworms did not accumulate the specific waste organics. Waste application reduced numbers and biomass of earthworms and numbers and kinds of microarthropods; however, both types of soil biota were able to recover.

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FOREWORD

EPA is charged by Congress to protect the Nation's land, air, and water systems. Under a mandate of national environmental laws focused on air and water quality, solid waste management and the control of toxic substances, pesticides, noise, and radiation, the Agency strives to formulate and implement actions which lead to a compatible balance between human activities and the ability of natural systems to support and nurture life.

The Robert S. Kerr Environmental Research Laboratory is the Agency's center of expertise for investigation of the soil and subsurface environment. Personnel at the Laboratory are responsible for management of research programs to: (a) determine the fate, transport and transformation rates of pollutants in the soil, the unsaturated zone and the saturated zones of the subsurface environment; (b) define the processes to be used in characterizing the soil and subsurface environment as a receptor of pollutants; (c) develop techniques for predicting the effect of pollutants on ground water, soil and indigenous organisms; and (d) define and demonstrate the applicability and limitations of using natural processes, indigenous to the soil and subsurface environment, for the protection of this resource.

This project was initiated to determine: (a) the loss and accumulation of oily waste constituents when applied to land in the cool and humid northeast, (b) the impact of single high waste-soil loading rates, and (c) the impact of waste application of soil biota. Results indicate that organic constituents would be lost and metal constituents would accumulate in the zone of incorporation at all of the loading rates used during the study. Both the loss and accumulation rates varied for different constituents. The soil biota was impacted at all loading rates; however, the degree of impact and time for recovery was dependent on the waste application rate. This information should prove useful to those responsible for regulating, designing, operating and monitoring industrial waste land treatment systems.

Clinton W. Hall

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Director
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ABSTRACT

The land treatment of an industrial oily waste was investigated to determine the loss and immobilization of constituents in the waste and to determine the impact of the waste and the application process on the soil biota. The project was a field study with laboratory components.

The waste was applied to field plots of a moderately to slowly permeable heavy silt loam in New York. The field plots consisted of four replicates each of natural controls, rototilled controls, and low, medium and high application rate plots. Wastes were applied in June 1982, October 1982 and June 1983. In June 1983, the plots that had received the low applications received a very high application and became the very high application plots. During the study, the waste was applied to the test plots at seven waste application rates that ranged from 0.17 to 9.5 kg total oil and grease/meter^2 or from 0.09% to 5.25% oil and grease in the zone of incorporation.

The application of the wastes increased the pH and volatile matter of the soils. Over the period of the study, the half life of the total oil and grease in the field plots ranged from about 260 to about 400 days. Not all of the applied oil was lost from the plots. The refractory fraction ranged from 20% to an apparent 50% of the applied oil and grease. The refractory fraction did not appear to adversely affect the soil biota.

Napthalenes, alkanes and specific aromatics were lost from the soil rapidly, especially in the warmer months. The half life of these compounds generally was less than 30 days.

The waste applications increased the concentration of several metals in the upper 15 cm of the soil. Except for sodium, all of the metals were immobilized in the upper 15 cm of the plots.

Earthworms bioaccumulated cadmium, potassium, sodium and zinc. The accumulation could not be related to waste application rates and occurred in worms from the control plots as well as in worms from the plots that received the wastes. The land treatment of these wastes did not cause any unexpected bioaccumulation of metals in the earthworms. The earthworms did not accumulate napthalenes, alkanes or specific aromatics that were in the applied waste.

Rototilling and waste application reduced the numbers and biomass of earthworms and the numbers and kinds of microarthropods in the field plots. Both types of soil biota were able to recover from the rototilling and waste application.

This report was submitted in fulfillment of Cooperative Agreement CR-809285 between Cornell University and the U.S. Environmental Protection Agency. This report covers the period of September 1981 through September 1984. All field and laboratory work was completed as of August 1984.

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SECTION 1

INTRODUCTION

BACKGROUND

Land treatment is a managed waste treatment and ultimate disposal process that involves the controlled application of a waste to a soil. The wastes are applied to the surface or mixed with the upper zone (0-1 ft. (0-0.3 m)) of soil. The objective of land treatment is the biological degradation of organic waste constituents and the immobilization of inorganic waste constituents. In this way, the assimilative capacity of the soil is utilized for waste management. Municipal wastewaters and sludges as well as industrial wastes can be treated using this process.

Land treatment should not be confused with: (a) the indiscriminate dumping of waste on land, (b) landfills, (c) deep well injection or (d) arid region waste impoundments. The design goals, long term impact, and degree of treatment of these other terrestrial systems are different from those of land treatment.

The performance of a land treatment site is a function of the dynamic physical, chemical, and biological processes that occur in the soil. As a result, the applied wastes are degraded, transformed and/or immobilized. These processes are similar to those that occur in conventional municipal and industrial waste treatment systems. One major difference is that with land treatment, the processes occur in an unconfined reactor filled with soil while with conventional systems the processes occur in tanks. Another major difference is that the rates of the physical, chemical and biological processes generally are slower than those in conventional systems. On the other hand, greater time is available for the reactions to occur at a land treatment site.

The design and operation of a land treatment facility is based on sound scientific engineering principles as well as on field experience. A land treatment site is designed and operated to: (a) maximize waste degradation and immobilization; (b) minimize release of volatile organic compounds; (c) minimize percolation of water soluble waste components; and (d) control surface water runoff. The managerial controls at land treatment sites are limited primarily to the application rates and the opportunity for cultivation (tilling), for moisture control (irrigation) and for nutrient additions. Temperature, climate conditions, and contact times are not control parameters.

The land treatment of industrial wastes is receiving increasing attention as a cost effective and environmentally sound method of waste management. Land treatment has been used as a waste management technology by petroleum refineries in the United States for more than 25 years. The technology also has been used by the exploration and production sector of the petroleum industry and for the cleanup of oil spills. About 100 land treatment facilities exist in the U.S. petroleum industry (1). In addition 26 of 38 Canadian refineries and at least ten refineries in Europe use land treatment (2).

The major concerns when land treatment is used for industrial wastes are the transformations, transport and fate of potentially toxic metals and organics that may be in the wastes. To date, feasible waste application rates have been based on: (a) physical and chemical characteristics of the soil such as permeability, cation exchange capacity (CEC), and pH; (b) mobility and plant uptake of constituents in the applied wastes; (c) the characteristics of the waste; and (d) the degradation and immobilization of constituents in the wastes.

Except as part of organic degradation, the soil biota rarely have been included in any research or full scale land treatment system or monitoring programs. However, the top layer of soil contains myriad microbes and invertebrates that degrade and transform the applied organics and that can affect the immobilization of the applied inorganics. To ignore the impact of the soil biota at a land treatment site is to ignore: (a) a major factor that may affect the performance of the site; and (b) the impact, such as bioaccumulation of potentially toxic compounds, that the applied wastes may have on the soil ecosystem.

The possible bioaccumulation of potentially toxic chemicals when wastes are applied to land is a continuing concern. As identified in the Resource Conservation and Recovery Act (RCRA), land disposal methods are to be protective of human health and the environment. The factors to be taken into account in assessing such protection are the persistence, toxicity, mobility and propensity to bioaccumulate of hazardous wastes and their constituents.

Earthworms are active indigenous soil invertebrates that assist the degradation of organic compounds. In addition, in the terrestrial food chain, earthworms represent one of the first levels of bioaccumulation that can occur when industrial wastes are applied to the land. It is appropriate to consider earthworms as a test organism to determine the impact of industrial waste on soil biota when land treatment is used for such wastes.

Microarthropods, such as mites and springtails, also are soil biota that are found in abundance in most soils and are secondary decomposers and detritus feeders. Studies have shown that they are affected adversely by insecticides and other chemicals added to the soil.

Both earthworms and microarthropods may be useful as indicators of the adverse effect of waste application to soil because they exhibit a series of effects in response to stressful situations. These are death, decreased growth and/or reproduction, and movement away from a stressful environment.

OBJECTIVES AND SCOPE

The overall purpose of this project was to determine: (a) the loss and immobilization of constituents of an oily waste when the waste was applied to the soil at different application rates, (b) the impact of the waste and the application process on the soil biota and (c) the general assimilative capacity of a soil when industrial wastes are land applied. The specific objectives were to evaluate:

(a) the loss of constituents of an industrial type waste when the waste was applied to land in the cool and humid northeast;

(b) the accumulation of waste constituents in the soil and their bioaccumulation in earthworms when the wastes were applied at varying rates;

(c) the impact of single, possibly large applications, rather than continuous applications, when such wastes are land treated or when there is an accidental application or spill of such wastes to the soil;

(d) the effect of an industrial type waste on soil biota such as earthworms and microarthropods.

The purpose of the project was to obtain comprehensive data that can be used to improve the design and operating criteria for industrial waste land treatment systems. The project was a field study with laboratory components.

SECTION 2

CONCLUSIONS

The objectives of this study were attained. The results indicated that the soil has the capacity to treat wastes such as those used in this study. Many of the organics in the applied waste were removed (lost) and the metals were immobilized when the wastes were applied to the soil intermittently and at varied application rates. The soil cultivation method (rototilling) and the applied waste had an immediate adverse impact on the soil biota (earthworms and microarthropods). However, the soil biota did recover with time. A fraction of the applied oil and grease was not removed during the study. The remaining organics and the metals did not seem to have any permanent adverse effect on the soil biota.

In addition, the application of these oily wastes to the field plots: (a) increased the pH of the acid soils (as much as one pH unit for the higher applications), (b) increased the temperature of the soil in the field plots that received the higher applications by 1 to 5°C, and (c) increased the organic matter of the soil by 1 to 5%.

The loss of organics applied to the soil varied. The loss of specific organics (napthalenes, alkanes and certain aromatics) in the field plots was rapid, especially in the warmer months. The half-life of these compounds generally was less than 30 days. In comparison, the half-life of the total oil and grease in the field plots ranged from about 260 to about 400 days. The oil and grease losses could not be correlated to the soil temperature, to other soil parameters, to the amounts of waste that were applied, or to the waste application rates.

All of the applied organics were not lost from the soil during the period of the study. The separation and identification procedures used were not able to identify the type or structure of the residual organics that remained in the soil at the end of the study. However, based on laboratory studies using soil from the field plots and the fact that both earthworms and microarthropods could repopulate the soil of the plots receiving the wastes, the organics remaining in the soil did not appear to result in a permanent adverse impact to the soil biota.

As a result of the waste applications, the concentration of many of the metals in the waste increased in the top 15 cm of the plots. This increase was especially noticeable as a result of the high and very high applications. However analyses indicated that, except for sodium in the very high application plots, at all of the other application rates, sodium and the other metals were immobilized in the top 15 cm of the soil.

The data indicated that soil biota such as earthworms and microarthropods can recover from intermittent applications of an oily waste. With time, the numbers and kinds of soil biota in the plots to which the wastes were applied could again become similar to those in the control plots, although at a rate not presently predictable.

The earthworms in the field plots did bioaccumulate several metals that were in the applied waste: cadmium, potassium, sodium and zinc. However, when the level of bioaccumulation was compared to data from other studies and to bioaccumulation in worms found in the control plots, it was apparent that the land treatment of these oily wastes did not cause any unexpected bioaccumulation of metals in the worms. The earthworms did not bioaccumulate napthalenes, alkanes or specific aromatics that were in the applied waste. Thus, the land treatment of these wastes did not lead to any bioaccumulation of waste constituents that was of apparent concern.

SECTION 3

EXPERIMENTAL PROCEDURES

GENERAL

This project was a cooperative agreement between Cornell University and the Robert S. Kerr Environmental Research Laboratory (RSKERL) of the Environmental Protection Agency (EPA). The research was conducted in laboratories of the Department of Agricultural Engineering, College of Agriculture and Life Sciences, Cornell University, and on land adjacent to the Cornell campus. The identification of the numbers and type of microarthropods in soil samples was done by Dr. Roy A. Norton of the Department of Environmental and Forest Biology, College of Environmental Science and Forestry (CESF), State University of New York, Syracuse, New York.

WASTE CHARACTERISTICS

The wastes used in this study were obtained with the help of RSKERL personnel from a site in Oklahoma on three separate occasions. The wastes were of unknown origin but were black, viscous, and were collected from the bottom of a lagoon that had been used to store wastes from oil refineries. The characteristics of the wastes applied to the field plots on the three dates discussed in this report are presented in Tables 1 and 2. In this report, the wastes used in the study are identified as oily wastes.

Although the wastes were collected from a large holding lagoon on three different occasions and it was unlikely that the contents of the lagoon were homogenous, the characteristics of the wastes were reasonably similar (Tables 1 and 2) especially when expressed on a moisture free basis. The water content of the three wastes did differ (Table 1).

The wastes were applied to the field plots to obtain a specific oil content in the soil of different plots. Samples of the wastes were analyzed prior to each application date and the oil data used to determine the volumes of a waste that were to be added to a specific plot. The higher water content of the wastes applied in October 1982 resulted in greater volumes of the waste being applied to achieve the desired oil content.

The wastes contained high concentrations of several metals (Table 2). Metals such as cadmium and nickel, which can be of concern at land treatment sites, were in low concentration.

The oily wastes had been contained in the holding lagoon for several years before the required quantities were removed and transported to the field site for application. Many volatile compounds may have been lost during the time the wastes were held in the lagoon.

TABLE 1. AVERAGE CHARACTERISTICS OF THE OILY WASTES APPLIED TO THE FIELD PLOTS

Parameter	Application Date June 1982	 October 1982	 June 1983
Water, %WB*	59.0	62.3	48.7
Ash, %MFB**	26.9	30.1	30.2
Oil & Grease, g/kg MFB	660	614	470
Total Kjeldahl Nitrogen, mg/kg MFB	2360	2320	2080
Total Phosphorus, mg/kg MFB	2620	ND***	1760
Chemical Oxygen Demand, g/kg MFB	1340	1250	1460
pH	7.2	7.1	6.7

*WB = wet basis, **MFB = moisture free basis, ***ND - not determined.

Several samples of the oily waste were analyzed by RSKERL to determine the type of organic compounds that were in the waste. Table 3 summarizes the compounds that were identified. The identification was accomplished by the GC/MS methods that were used at RSKERL.

PCB analyses indicated that if any of the following were present, Arochlor 1221, 1016/1242, 1254, or 1260, the concentrations were less than the detection limit of 0.75 micrograms/gram.

FIELD SITE

The land used for application of the waste was an old field site in Tompkins County, New York, near Cornell University. The site had not been used for agricultural purposes and had not received applications of lime, fertilizer, pesticides or herbicides for over 10 years prior to use in

TABLE 2. AVERAGE CONCENTRATIONS OF METALS IN THE OILY WASTE APPLIED TO THE FIELD PLOTS, mg/kg MFB*

	Application Date		
Metal	June 1982	October 1982	June 1983
Al	7320	12200	10700
Ca	73200	75400	78000
Cd	1.7	2.0	1.8
Cr	730	780	770
Cu	140	150	164
Fe	6100	8960	9160
K	1630	2660	2240
Mg	2050	2760	2340
Mn	110	147	146
Na	2150	2880	1670
Ni	15	22	21
Pb	160	206	253
Ti	90	97	179
V	11	18	17
Zn	1850	1840	1780

* = moisture free basis

TABLE 3. ORGANIC COMPOUNDS IN THE OILY WASTE APPLIED TO THE FIELD PLOTS[+]

I.

Nonane
Decane
Undecane
Dodecane
Tetradecane
Pentadecane
Hexadecane
Heptadecane
Octadecane
Nonadecane
Eicosane
Heneicosane
Docosane
Tricosane
Tetracosane
Pentacosane

II.

"Branched alkanes"

III.

Dimethylhexane
Dimethyloctane
Dimethylnonane
Dimethylundecane
Ethylundecane
Cyclohexylundecane
Methylpropyldodecane
Oxybisdodecane

IV.

Methylcyclopentane
Propylcyclohexane

V.

Dimethylbenzene
Trimethylbenzene
Tetramethylbenzene
Ethylmethylbenzene
Dimethylethylbenzene
Ethenylethylbenzene
Ethylpropenylbenzene

VI.

Naphthalene
1-methylnaphthalene
2-methylnaphthalene
Dimethylnaphthalene
Methylethylnaphthalene
Trimethylethylnaphthalene
Tetrahydronaphthalene
Dihydroacenaphthalene
Isocyanatonaphthalene

VII.

Methylphenanthrene
Dimethylphenanthrene
Trimethylphenanthrene

VIII.

Dihydromethylindene
Dimethylbiphenyl

[+] Determined by RSKERL personnel using GC/MS techniques.

this project. The site had been mowed annually to hinder growth of woody plants.

The USDA Soil Conservation Service Soil Survey for Tompkins County identifies the soil at the site as Rhinebeck silt loam. This soil has about one foot of moderately to slowly permeable heavy silt loam over slowly permeable silty clay loam or silty clay. The lower layer extends to a depth of 2 to 3 feet and is underlain by layers of silty clay separated by thin layers of silt. The soil is somewhat poorly drained and exists on nearly level to moderate slopes in glacial lake areas.

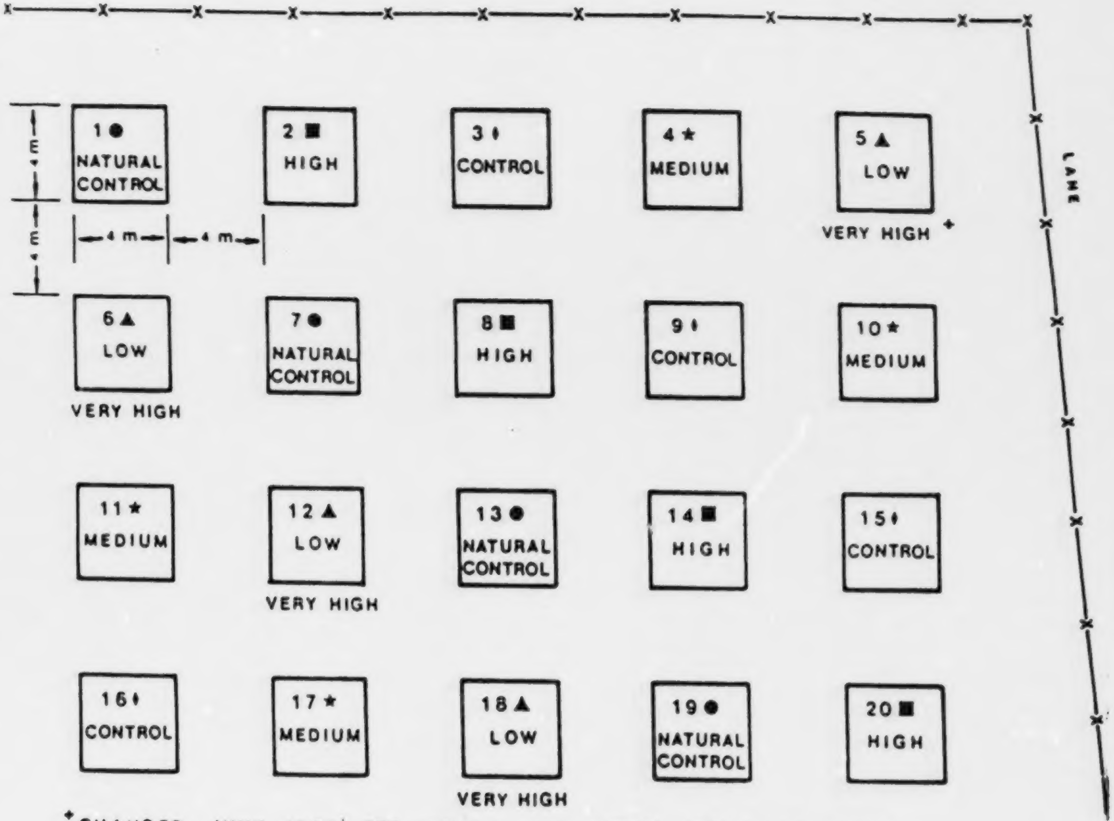
The field site (Figure 1), consisted of 20 plots, 4 meters by 4 meters, with 4 meters of border area surrounding each plot. Four waste application rates plus natural and rototilled controls were used at the site. There were four replications for each waste application rate and type of control. All plots were mowed prior to each waste application. All plots, except the natural controls, were rototilled after each application of the waste. The four rototilled control plots had no waste applied but were rototilled. The four natural control plots had no rototilling or oily waste applied and were used to separate the effects of the rototilling and the waste applications. The applied wastes were distributed over the plot surface as uniformly as possible, and were then rototilled into the soil to a depth of about six inches (15 cm). Thus the zone of incorporation for these plots was the top six inches.

Each test plot (16 m^2) was marked with corner stakes to permit placement of a framed grid to define 400-0.04 m^2 (20 cm x 20 cm) sampling subplots (Figure 2). Three different subplots were sampled on each sampling date to determine changes in incorporation zone characteristics and in earthworm and microarthropod populations. To eliminate edge effects, the edge subplots were not sampled. The subplots that were sampled from among the 324 possibilities were determined using a random number table. Thus different sampling locations were used at each plot each time samples were taken. No subplot was sampled twice during the study. Examples of the subplots that were sampled on the noted sampling days are included in Figure 2. An elevated plank platform was used to obtain the samples so that the plots were not disturbed or contaminated while the samples were taken. The project personnel did not walk on the plots except during the waste application and the rototilling.

Natural vegetation such as grass was allowed to become re-established on the plots in the months after the waste application.

ANALYTICAL PROCEDURES

Soil samples were taken from each plot at approximately monthly intervals except during the winter months. Core samples were taken from the top 15 cm (six inches) of the plots. This depth represented the depth to which the wastes were added and mixed. At each sampling, three soil cores were taken from the randomly determined subplots. Hand sorting was used to determine earthworm numbers and biomass from each core. Prior to

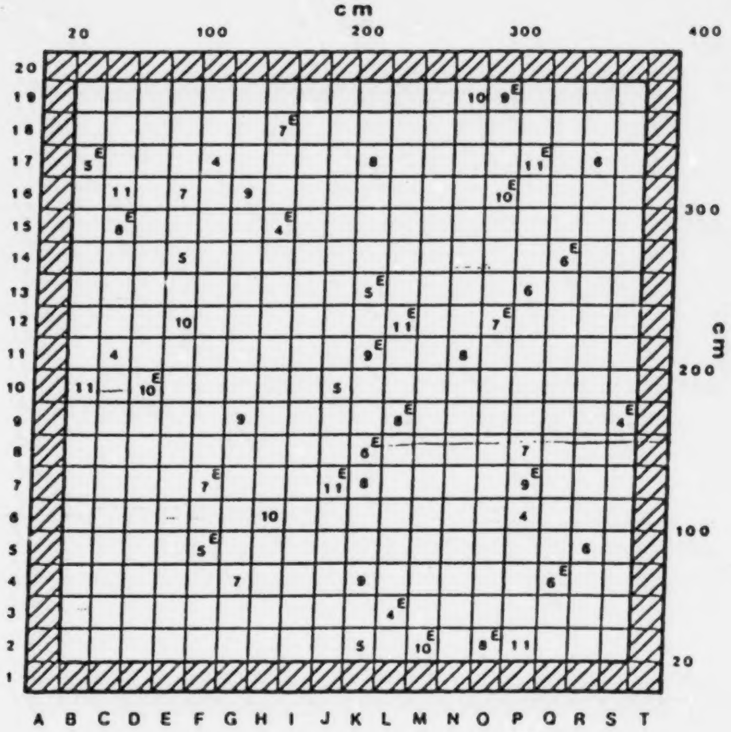

LANE
4 m
4 m
4 m
4 m
1 ● NATURAL CONTROL
2 ■ HIGH
3 ♦ CONTROL
4 ★ MEDIUM
5 ▲ LOW
VERY HIGH +
6 ▲ LOW
VERY HIGH
7 ● NATURAL CONTROL
8 ■ HIGH
9 ♦ CONTROL
10 ★ MEDIUM
11 ★ MEDIUM
12 ▲ LOW
VERY HIGH
13 ● NATURAL CONTROL
14 ■ HIGH
15 ♦ CONTROL
16 ♦ CONTROL
17 ★ MEDIUM
18 ▲ LOW
VERY HIGH
19 ● NATURAL CONTROL
20 ■ HIGH

+CHANGED JUNE 1983; SEE TABLE 7 FOR ACTUAL APPLICATION RATES

FIGURE 1
SCHEMATIC OF THE PLOTS USED IN THE FIELD STUDY

EXAMPLE OF SUBPLOTS SAMPLED ON SPECIFIC SAMPLING DATES

	APRIL-4			MAY-5			JUNE-6			JULY-7		
MICRO-ARTHROPODS	F-17	C-11	P-6	E-14	J-10	K-2	R-17	P-13	R-5	E-16	P-8	G-4
EARTHWORMS	H-15	S-9	L-3	B-17	K-13	F-5	Q-14	K-8	Q-4	H-18	O-12	F-7


cm
20
100
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400
300
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100
20
cm
A B C D E F G H I J K L M N O P Q R S T
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20

▨ – INDICATES EDGE PLOTS NOT TO BE SAMPLED

FIGURE 2
SUBPLOTS IN EACH TEST PLOT USED FOR SAMPLING

measuring the physical and chemical characteristics of the soil, the cores from each plot were composited. Residual soil was returned to the plots and used to fill in the core holes.

The microarthropod samples were soil cores approximately 6 cm in diameter and 6 cm deep. The microarthropods were separated from the soil by inverting the soil core in a heat-gradient extractor for one week. The upper half of the soil core was heated by light bulbs and the lower half exposed to the ambient temperature in a 5°C environmental chamber. The microarthropods followed the humidity gradient that was established and fell from the bottom of the funnel into collecting jars of ethanol preservative. The type of microarthropods were identified and counted. Greater details about the microarthropod extraction and identification procedures are presented in Appendix A.

Metals and certain organics in the waste, soil and earthworm samples were analyzed by personnel at the EPA Robert S. Kerr Environmental Research Laboratory (RSKERL). Cornell personnel analyzed the waste and soil samples for more routine parameters. Dr. Norton (CESF) counted and identified the microarthropods.

The analyses conducted by RSKERL and Cornell personnel are noted in Table 4. The methods used to analyze the waste and soil samples at Cornell are noted in Table 5. In addition, Cornell personnel counted and identified the earthworms.

Soil, waste, and earthworm samples were analyzed for metals and specific organics at RSKERL. Dried samples of the soil and earthworms and liquid samples of the waste were sent to RSKERL for metal analysis. For organic compounds, methylene chloride extracts of the soil and earthworms were sent to RSKERL for analysis. The extracts were prepared by mixing 10 grams of wet sample with anhydrous sodium sulfate and extracting the mixture for two hours or at least 20 cycles with methylene chloride. This extraction method was supplied to the Cornell investigators by the RSKERL project officer. The methods used to prepare the soil and worm samples for analysis by RSKERL are presented in detail in Appendix B.

The impact of the waste on earthworms was evaluated in the laboratory using the general methodology outlined in Figure 3. The contact and artificial soil tests were developed and tentatively approved by the European Economic Community (EEC) (7). The growth and reproduction tests have been developed by Cornell personnel, have been evaluated with many chemicals and wastes, and have been used in other research studies. With each of the earthworm tests, controls were included. The details of these methods are presented in SECTION 4.

A quality assurance plan was prepared by the Cornell investigators and approved by the RSKERL project officer in early 1982. That plan identified sampling procedures, quality control checks, and procedures to assess data precision and accuracy.

TABLE 4. FIELD STUDY MEASUREMENTS

Samples	Parameters Measured	Laboratory That Performed Analysis
I. Oily Waste	total solids	Cornell
	volatile solids	Cornell
	COD	Cornell
	TKN	Cornell
	total phosphorus	Cornell
	pH	Cornell
	oil	Cornell
	metals	RSKERL
	specific organics	RSKERL
II. Earthworms	metals	RSKERL
	specific organics	RSKERL
III. Field Test Plot Soils	metals	RSKERL
	specific organics	RSKERL
	CEC	Cornell
	total solids	Cornell
	volatile solids	Cornell
	pH	Cornell
	TKN	Cornell
	oil and grease	Cornell

TABLE 5. METHODS USED FOR ANALYSIS OF WASTE AND SOIL SAMPLES

		Sample	
Parameter	Method	Waste	Soil
Moisture (Total Solids)	Standard test method for water in petroleum products and bituminous materials by distillation - ANSI/ASTM D95-70*	X	
	Total residue dried at 103-105°C - *Standard Methods*†, 209A. and *Methods of Soil Analysis*§, 7-2.2.		X
Ash	Standard test method for ash from petroleum products - ANSI/ASTM D482-80*	X	
Volatile Solids	Total volatile and fixed residue - *Standard Methods*†, 209 E		X
Total Kjeldahl Nitrogen (TKN)	Semi-micro Kjeldahl technique - McKenzie and Wallace (1954) and *Standard Methods*†, 420 B.	X	X
Total Phosphorus	Sulfuric acid - nitric acid digestion - *Standard Methods*, 424 C II followed by Vanadomolybdophosphoric acid colorimetric method - *Standard Methods*†, 424 D.	X	
Chemical Oxygen Demand	Dichromate reflux method, *Standard Methods*†, 508 A.	X	
Oil and Grease	Extraction method for sludge samples - *Standard Methods*†, 502 D.	X	
pH	Glass electrode - *Standard Methods*†, 423	X	
	Measurement in 0.01 M $CaCl_2$ solution - *Methods of Soil Analysis*§, 60-3.5		X
Temperature	Bimetallic thermometer - *Methods of Soil Analysis*§, 2-2.3		

*Reference 5
†Reference 3
§Reference 4

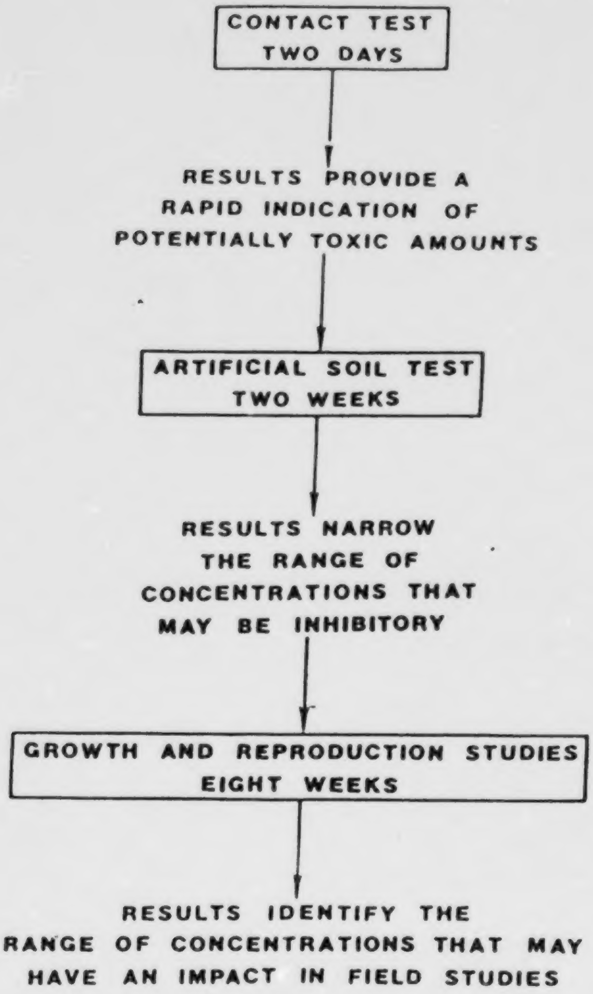

CONTACT TEST
TWO DAYS
RESULTS PROVIDE A RAPID INDICATION OF POTENTIALLY TOXIC AMOUNTS
ARTIFICIAL SOIL TEST
TWO WEEKS
RESULTS NARROW THE RANGE OF CONCENTRATIONS THAT MAY BE INHIBITORY
GROWTH AND REPRODUCTION STUDIES
EIGHT WEEKS
RESULTS IDENTIFY THE RANGE OF CONCENTRATIONS THAT MAY HAVE AN IMPACT IN FIELD STUDIES

FIGURE 3
METHODOLOGY USED TO DETERMINE THE
IMPACT OF WASTES ON EARTHWORMS IN THE LABORATORY STUDIES

In addition, samples of a freeze-dried, digested municipal sludge standard were supplied from EPA-Cincinnati through the RSKERL project officer to Cornell. Such samples were used to determine the precision of the analyses being run at Cornell (Table 4). The analyses for all parameters were very close to the mean values identified by EPA for the standard sludge sample. Samples of the "standard" sludge were analyzed periodically as part of the routine monthly sampling and analysis. Results of these quality assurance analyses for oil and grease are presented in Appendix K.

At Cornell, for each waste sample and each parameter, at least five replicates were analyzed and used to determine a mean and standard deviation. For each soil sample and each parameter, at least two replicates were analyzed.

SPECIAL STUDIES

Two special studies were conducted to determine: (a) the variability in the characteristics of the soil samples taken from various locations in the field plots, and (b) the precision and accuracy of the analytical method used for oil and grease when used with soil samples. The spatial variability study identified the extent to which the variability of the data was due to the non-homogeneity of waste application and rototilling. The results of the oil and grease analytical method evaluation established the extent to which this method extracted the oil and grease in the waste and soil samples. These special studies and the results that were obtained are presented in Appendix C (spatial variability) and Appendix D (oil and grease analytical method).

SECTION 4

LABORATORY STUDIES

The laboratory studies determined the tolerance level of earthworms to the waste. The worms were Eisenia fetida, a worm used extensively in laboratory studies throughout the world. The three step program noted in Figure 3 provided an indication of the waste concentrations that had an impact on the worms.

The contact test method generally is the first method used for these purposes. The contact test attempts to determine the approximate lethal concentration for the waste being tested. A filter paper strip is inserted into a small (4 oz.) glass vial to cover the sides of the vial and a specified amount of a waste or chemical is evenly distributed over the filter paper. Due to the viscous nature of the oily waste, it was difficult to utilize small uniform samples in the contact test. Because of these problems, the results of the contact test were inconclusive.

The artificial soil test is used to quantify the toxic effect of the specific waste or chemical being tested. Because soils are a heterogenous mixture, an artificial soil containing, on a dry wet basis, a mixture of sand (69%), kaolinite clay (20%), ground peat (10%) and limestone (1%) is used in this test. The pH of the mixture is adjusted to 7.0 using the limestone, and the moisture content is adjusted to 35% using deionized water. The test containers are covered 125 mm x 65 mm dishes with 400 g (dry weight) of the test medium. There were four replicates for each waste concentration tested. Ten adult worms were used per test. The average weight of the worms was determined at the beginning and end of each test, which ran for 14 days. Worm survival also was recorded. The results are discussed later in this section.

Neither the contact or artificial soil test indicates the sublethal effects that a particular chemical might have on earthworm growth rates or reproduction. The growth and reproduction test is used to evaluate such effects.

In the growth and reproduction test, a specific quantity of waste was mixed with horse manure, a known earthworm food source. The resultant waste concentration was based on the oven dry weight of the food source. The mixture was placed on a layer of moistened soil in a 20 mm x 100 mm petri dish. Two E. fetida, less than 10 mg each, were added to each dish. Four replicates of each waste concentration were included. Four and six weeks after the experiment was initiated, the residual mixture was removed from the dishes and the worms were fed a fresh mixture that included the proper waste concentration. The worms were weighed at four, six, and eight weeks. These weights as well as the cocoon production, were compared to controls to determine the sublethal effects of the waste being tested. Results of these tests also are discussed later in this section.

These studies determined the impact of the wastes on the earthworm *E. fetida* under controlled laboratory conditions. The intent was to obtain an estimate of the impact: (a) before the liquid waste was applied and therefore to obtain some estimate of the application rate that should be used initially; and (b) so that the impact could be compared to data that would result from the field studies.

Neither the artificial soil or the growth and reproduction tests are identical to conditions that occur in a field land treatment site. In both tests, there is little opportunity for loss of volatile constituents. In addition the microorganisms in the media may not be acclimated to the waste and degradation may not be as rapid as it would be in the field. Worms other than *E. fetida* may exist at a field site. The media used in the artificial soil test approximates the conditions in a field soil reasonably well. However, the media used in the growth and reproduction test contain more organic matter and a greater cation exchange capacity (CEC) than exists in field soils. In spite of these differences, the results obtained using these tests can identify the relative impact of a waste when it is applied to the soil.

The results of the artificial soil test indicated that the oily waste can affect the survival of *E. fetida* (Figure 4). This impact was observed when the concentration of waste to which the worms were exposed exceeded about 1000 mg wet waste per kg of artificial soil (wet weight). This corresponded to about 420 mg of oil and grease per kg of artificial soil on a moisture free basis. The LC_{50} value was determined using the mehtod of Litchfield and Wilcoxon (40) and was 1540 mg of the wet waste per kg of the artificial soil. The 95% confidence interval values were 1360 and 1670 mg/kg.

Results obtained in the growth and reproduction tests are shown in Figure 5. At waste concentrations up to 20,000 mg wet waste per kg dry weight of horse manure, no decrease in worm weight occurred (Figure 5). Cocoon production per worm (Figure 5) and cocoon viability did not decrease until large quantities of the waste were incorporated in the manure.

The impact of the higher concentrations of the oily waste was abrupt and severe. At concentrations of 25,000 mg wet waste per kg dry weight of manure or greater, all the test worms died. For this waste, the concentration of 20,000 mg wet waste/kg dry manure was the equivalent of an oil concentration of about 5400 mg/kg dry manure or about 0.54% by weight.

The results of the growth and reproduction test were different (showed less impact) than those of the artificial soil test (Figure 4) with respect to worm mortality. The different characteristics of the

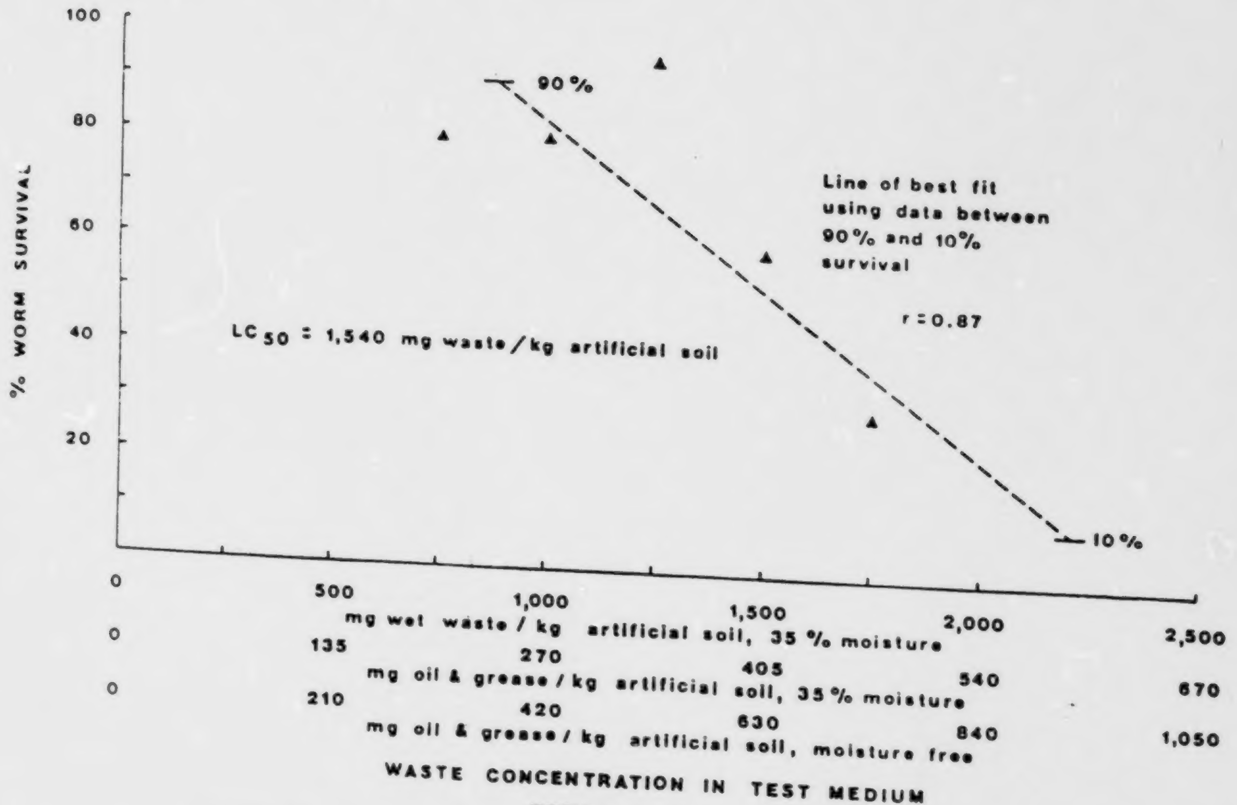


FIGURE 4
TOXICITY OF THE OILY WASTE IN THE ARTIFICIAL SOIL TEST

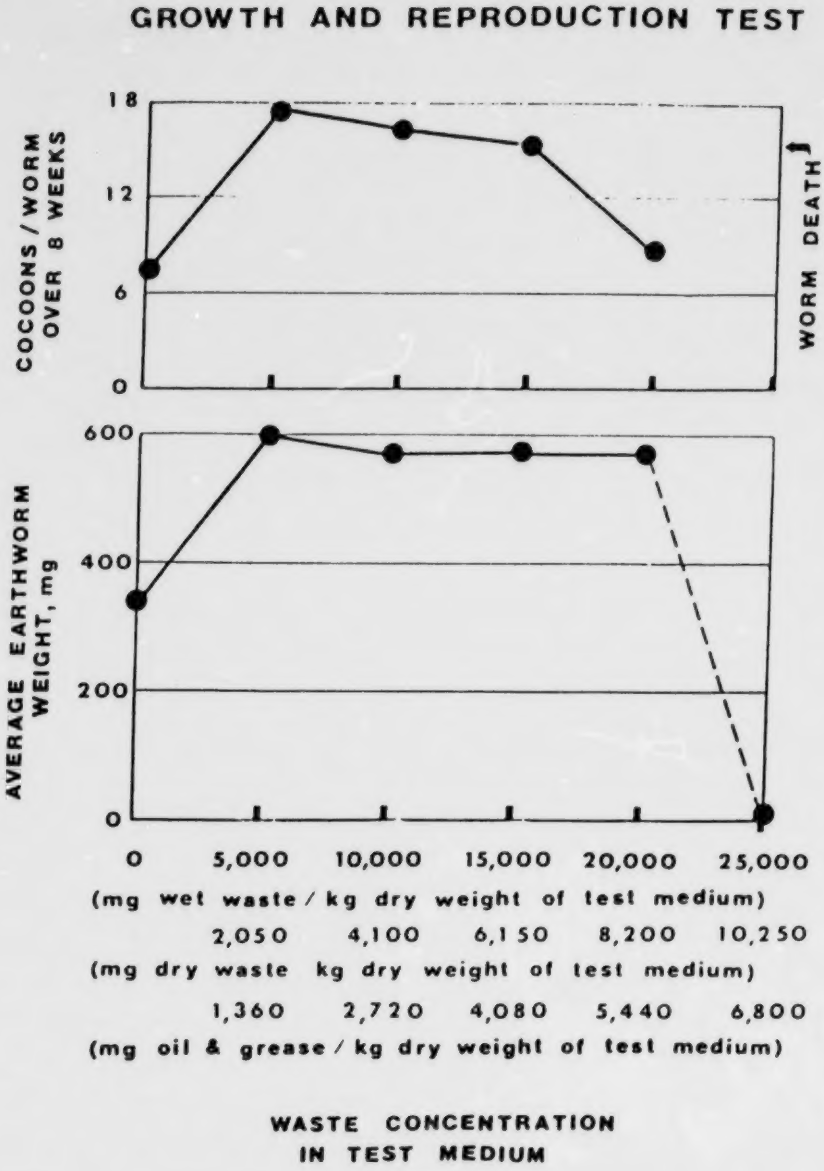


FIGURE 5
WORM BIOMASS AND COCOON PRODUCTION IN THE GROWTH AND REPRODUCTION TESTS

media in the two tests was one reason for the different results. The artificial soil test medium consists of sand, peat, kaolin and a small amount of limestone plus the waste. Microorganisms that may aid in decomposition of organics are only added with the wastes.

The growth and reproductive test medium is a mixture of horse manure and the waste source. This media is much higher in organic content and in indigenous microorganisms than the artificial soil test media. The greater organic content provides more sorption sites for components of the waste and the microorganisms can increase the decomposition of the organics in the wastes. Both factors could decrease the toxicity of the waste to the earthworms.

The artificial soil test method also was used with soil from several of the field plots to determine whether the waste-soil mixture had an adverse effect on the worms. In these experiments, soil from the zone of incorporation at the plots replaced the artificial soil. The results are presented in Table 6. For the medium and high plots (plots 11 and 14), the most recent waste application had been in October 1982. Thus the results show the effect of the residual waste after a nine to eighteen month period. For the very high plots, the most recent application had been in June 1983 and was the highest application rate used.

The worm survival data from Table 6 were compared (Figure 6) to the interval of time between when the waste was applied to the plot and when the soil samples were taken for these artificial soil analyses. Although the soils were taken from different plots and different waste applications had occurred, a general pattern is apparent. Greater worm survival resulted when there had been a longer interval of time since the waste had been applied.

Considerable loss of oil and grease occurred after the waste applications (SECTION 5). The lower molecular weight and more easily biodegradable organics were lost more rapidly. Therefore organic compound concentrations different from those in raw wastes were present in the soils used in these artificial soil tests. The total oil and grease content of the soils that were used in these tests are presented in Table 6.

Several inferences are apparent from the data in Table 6. First, the apparent adverse impact when using the field soils occurred at higher oil and grease concentrations than the impact that resulted from the oily waste in the previous artificial soil and growth and reproduction tests (Figures 4 and 5). However, it should be recognized that the oil and grease test is only a gross measure of the waste constituents in a soil or a waste and that biodegradation and volatilization had occurred in the field soils.

The second inference is that even nine to eighteen months after the October 1982 waste application, some residual adverse impact to the worms could be discerned (Figure 6). Third, there was an immediate adverse effect on the worms from the June 1983 very high application and all of the worms died. Four months later (October 1983), there was a less severe

TABLE 6. RESULTS OF THE ARTIFICIAL SOIL TEST USING SOIL OBTAINED FROM SPECIFIC FIELD PLOTS ON THE NOTED SAMPLING DATES

Plot Number	Plot Type	Percent of Initial Worm Weight*			Oil and Grease in the Noted Plots**		
		July 1983	October 1983	April 1984	July 1983	October 1983	April 1984
13	Natural Control	101	109	102	650	540	570
11	Medium[†]	87	92	98	5,100	5,720	4,990
14	High[†]	65	77	89	16,340	11,460	10,500
5	Very High[††]	0[+]	53	75	47,600	32,700	27,900
6	Very High[††]	0	59	81	59,200	35,100	29,100
12	Very High[††]	0	65	68	54,000	34,000	34,000
18	Very High[††]	0	60	72	62,600	35,000	35,600

*The weight of the worms after 14 days as a percentage of the weight of the worms at the beginning of the experiments.

**Average mg of oil and grease per kilogram of soil (MFB) in the soil from the plots that were used in the artificial soil test.

[+] A value of zero indicates that all the worms died.

[†] Previous waste application occurred in October 1982.

[††] Previous waste application occurred in June 1983.

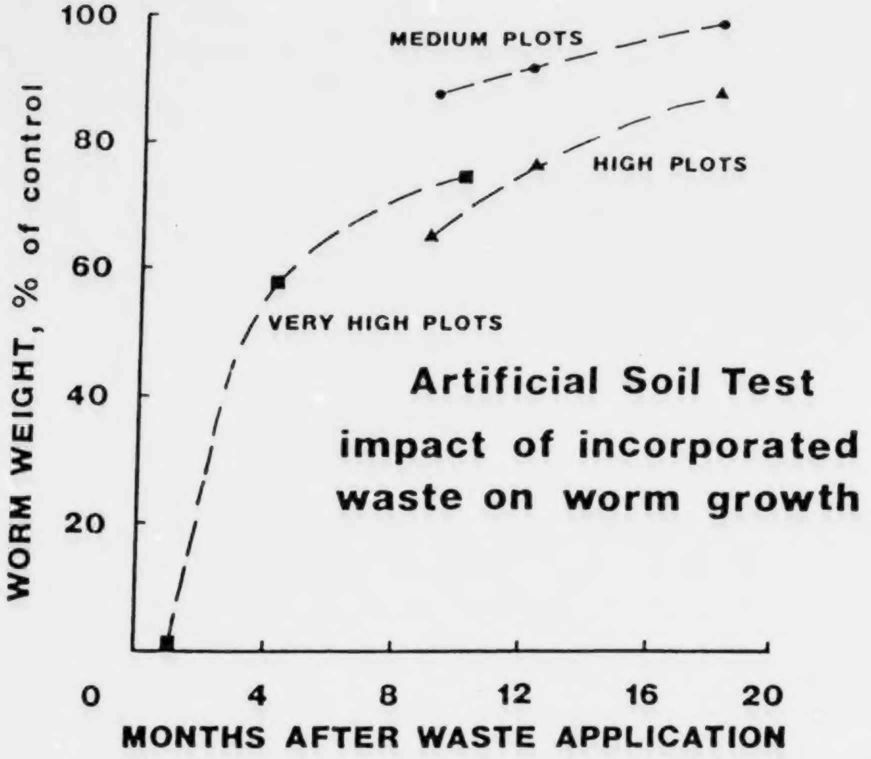


FIGURE 6
THE IMPACT OF SOIL FROM THE FIELD PLOTS ON EARTHWORM SURVIVAL USING THE ARTIFICIAL SOIL TEST

impact and even less of an impact ten months later (April 1984). The applied waste became less toxic to the worms with time, again probably due to the degradation and loss of components of the applied oily waste.

As is discussed in SECTION 5, there were many factors that affected the number and biomass of worms in the field plots--temperature, moisture content, rototilling, vegetation or lack of it, and the applied waste. The artificial soil test data in Table 6 does identify the immediate and lingering impact of the applied waste and adds to the understanding of how the changes that took place in the field plots affected the earthworms.

The results from the artificial soil tests using the field soils demonstrate that: (a) oily wastes of the nature used in this study can adversely affect the earthworms in the soil, and (b) as the organic compounds are lost from the soil, the adverse impact is decreased. These results were verified by data on earthworm numbers and biomass obtained from the plots during the study (SECTION 5).

SECTION 5

FIELD STUDY

WASTE APPLICATION

The oily waste was applied to specific test plots in June 1982, October 1982, and June 1983. These were the times when the wastes were able to be obtained from Oklahoma. The characteristics of the wastes applied on these dates have been presented in Tables 1, 2 and 3. The field plots were identified in Figure 1.

In October 1982, the low, medium and high plots received larger application rates than were applied in June 1982. In June 1983, a very high waste application was made to field plots 5, 6, 12 and 18, the plots that had received the initial low application rates. Thus the effect of seven application rates, ranging from 0.17 to 9.5 kg oil per meter^2 of surface area was evaluated. The application rates for the respective plots are noted in Table 7. The rates spanned the range likely to be used under actual field conditions.

Only the indigenous nutrients and trace elements in the soil and the waste were available to the micro- and macroorganisms as the wastes were degraded. No fertilizers or other amendments were added to the plots.

The plots were only cultivated (rototilled) immediately before and after the wastes were applied. No subsequent cultivation occurred to aerate the zone of incorporation. The plots were undisturbed after the combined waste applications and rototilling and only natural aeration occurred in the plots. This is different than what would occur at most industry land treatment sites where frequent tilling may occur to promote mixing and aeration and to increase degradation and other losses. This approach was taken in order to approximate the changes that would occur under conservative and non-optimum conditions such as when there may be single or highly intermittent waste applications or when a spill would occur. The approach also caused one less variable, the frequency and type of aeration (tilling), to be included in the study.

CLIMATIC DATA

Suitable environmental conditions, especially temperature and moisture content, are necessary for the soil biota and for degradation of the organic matter in the applied waste. The precipitation and soil temperature patterns that occurred at the field site are shown in Figures 7 and

TABLE 7. FIELD PLOT STUDY APPLICATION RATES, kg/m^2

Plot Number	June 1982			October 1982			June 1983		
	Wet Waste+	Dry Matter++	Oil & Grease	Wet Waste	Dry Matter	Oil & Grease	Wet Waste	Dry Matter	Oil & Grease
5	0.63	0.26	0.17	4.94	2.09	1.41	39.7	20.4	9.5
6	0.63	0.26	0.17	4.94	2.09	1.41	39.7	20.4	9.5
12	0.63	0.26	0.17	4.94	2.09	1.41	39.7	20.4	9.5
18	0.63	0.26	0.17	4.94	2.19	1.62	39.7	20.4	9.5
4	1.25	0.51	0.34	9.94	4.20	2.83	NA*	NA*	NA*
10	1.25	0.51	0.34	10.90	4.60	3.10	NA	NA	NA
11	1.25	0.51	0.34	9.94	3.98	2.52	NA	NA	NA
17	1.25	0.51	0.34	9.94	3.98	2.52	NA	NA	NA
2	2.49	1.02	0.68	19.80	7.76	4.46	NA	NA	NA
3	2.49	1.02	0.68	19.80	6.71	5.31	NA	NA	NA
14	2.49	1.02	0.68	19.80	6.91	4.74	NA	NA	NA
20	2.49	1.02	0.68	19.80	6.08	3.72	NA	NA	NA

*No application - waste only applied to plots 5, 6, 12, and 18 in June 1983.

+The quantities of wet waste applied were determined by weighing. A barrel of waste was weighed, the waste pumped out and applied, the weight of the barrel and any residue determined, and the amount applied to a plot determined by difference. Although every attempt was made to have each barrel applied on a given day contain the same material, it was not always possible. Some of the barrels contained different amounts of water and oil and grease. The data in this Table indicate the amounts that were added to the noted plots.

++Dry matter was determined by substracting the moisture content of the waste, measured using the method in Table 5, from the wet weight of the waste.

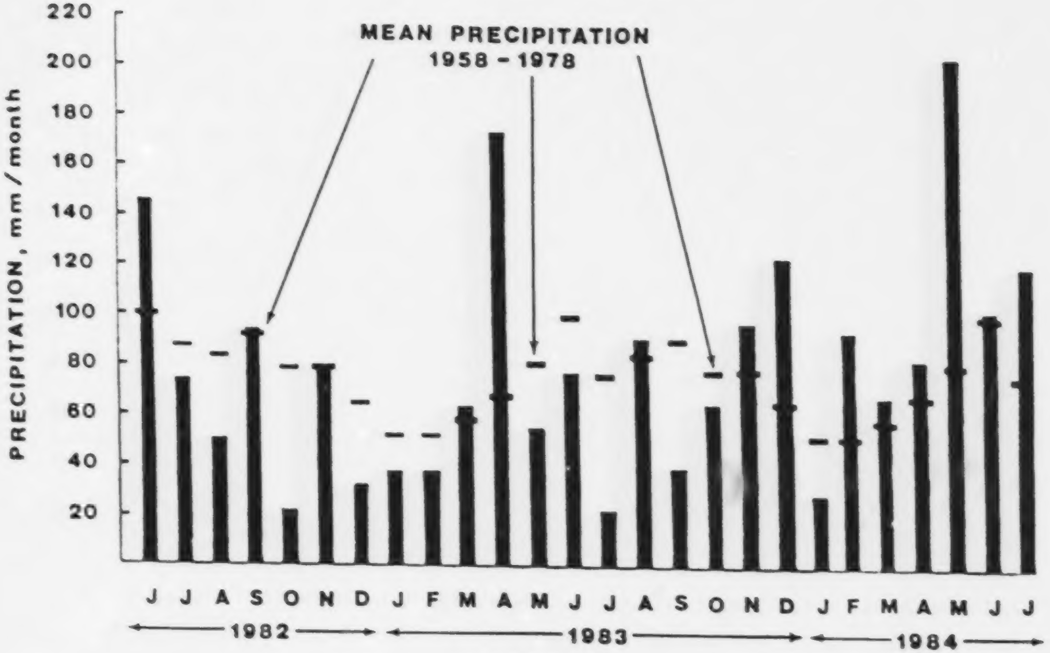


FIGURE 7
MONTHLY PRECIPITATION AT THE FIELD SITE

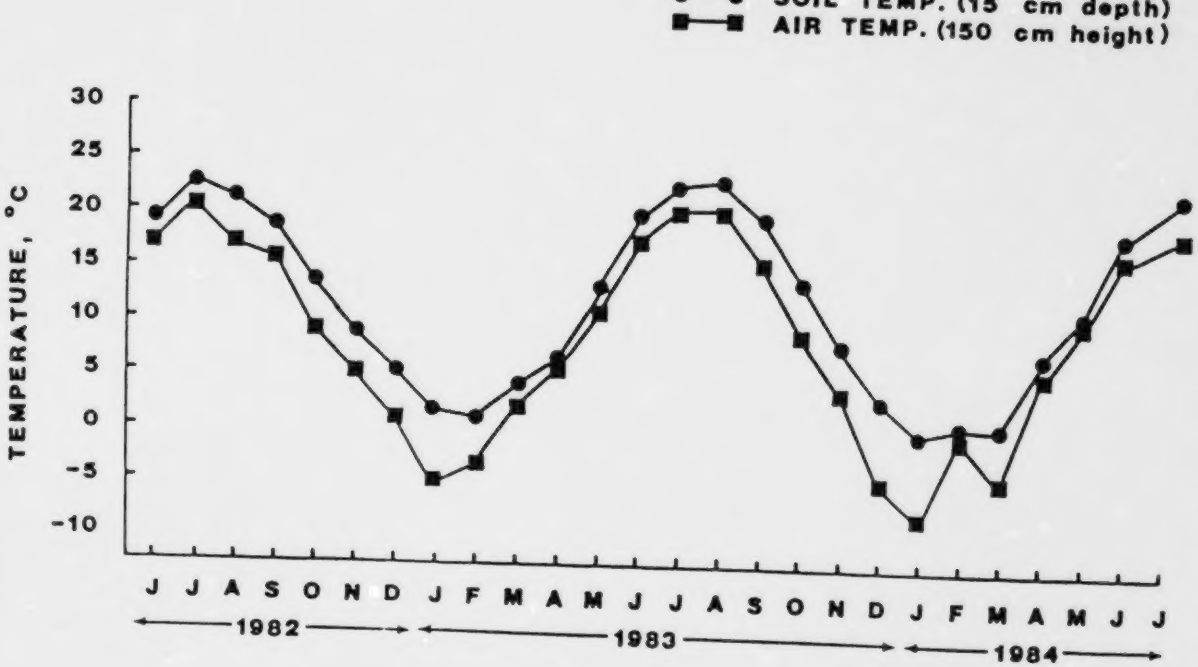


FIGURE 8
MONTHLY AVERAGE SOIL AND AIR TEMPERATURES AT ITHACA, NY

8. The summers of 1982 and 1983 were relatively dry as shown by the precipitation data (Figure 7). The winters of 1982-83 and 1983-84 were relatively mild and the upper 15 cm of the soil was frozen for only a short period of time during these winters. The average soil temperatures at a depth of 15 cm consistently were warmer than the average air temperature (Figure 8).

The data in Figure 8 represent average monthly data for an official weather station less than one-half mile from the field site. The soil temperatures measured at the 15 cm depth in the control field plots throughout the year were within 1 or 2°C of the soil temperatures at the official weather station. Thus, the soil temperature pattern noted in Figure 8 can be considered as representative of the pattern at the field site.

Because of wet and cold conditions during the latter months in 1983 and the spring of 1984, no soil samples were taken from the plots during the periods of December 1982 through February 1983 and November 1983 through April 1984.

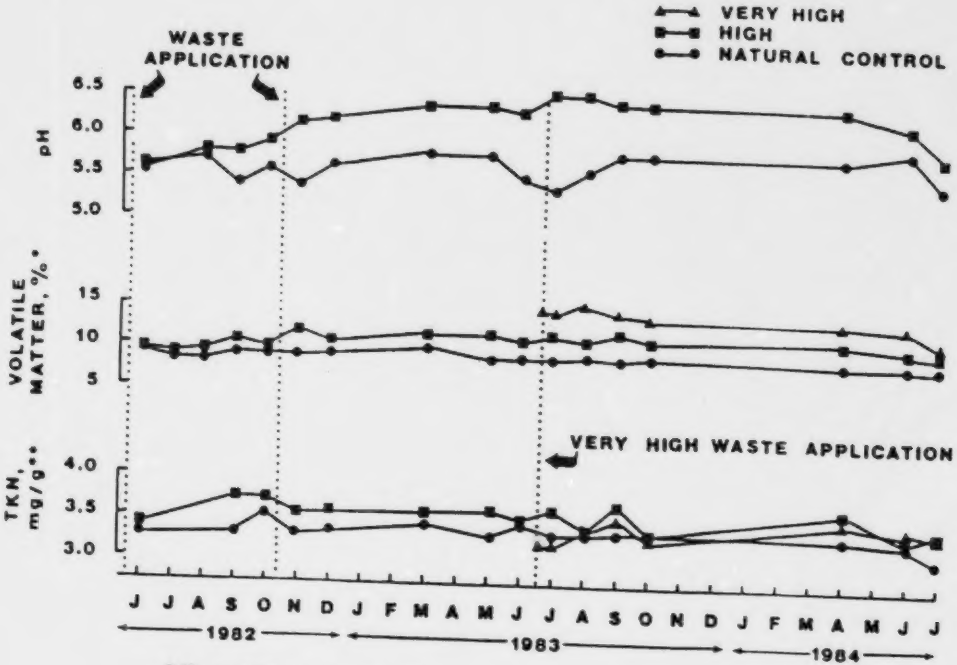
SOIL CHARACTERISTICS

General - Some of the soil characteristics changed as a result of the waste application. Figures 9 and 10 indicate typical results. For clarity, only the average values for the natural control, high and very high plots are presented. Also shown are dates when the waste was applied and when the soil samples were taken. The mean values for the characteristics of the plots receiving similar application rates are noted in Appendix F.

pH - The pH of the plots that received the high applications of the oil waste increased. The increase was pronounced for the plots that received the very high applications in June 1983 (Figure 10). With the very high application, the soil pH increased by more than one pH unit. After the waste applications, the pH stayed at above background levels during the rest of the study (Figures 9 and 10).

Nitrogen and Volatile Matter - There were no statistically significant increases in the nitrogen content of the soils (Figure 9) as a result of the waste applications. The TKN concentration in all of the plots was about 3.5 grams per kilogram of moisture free soil.

The volatile matter in the soil was increased by the waste applications (Figure 9). Until the waste applications in October 1982, the volatile matter in the plots was about 9% of the soil on a moisture free basis. After the October 1982 application, the volatile matter in the medium application plots was about 10% and in the high application plots was about 11%. After the application in June 1983, the volatile matter in the very high application plots was in the range of 14 to 15%. There appeared to be a slight decrease of the volatile matter in the very high application plots with time.



* % moisture free soil
** mg/g moisture free soil

FIGURE 9
MEAN VALUE OF pH, VOLATILE MATTER AND TKN IN THE SOIL OF THE FIELD PLOTS, n = 4

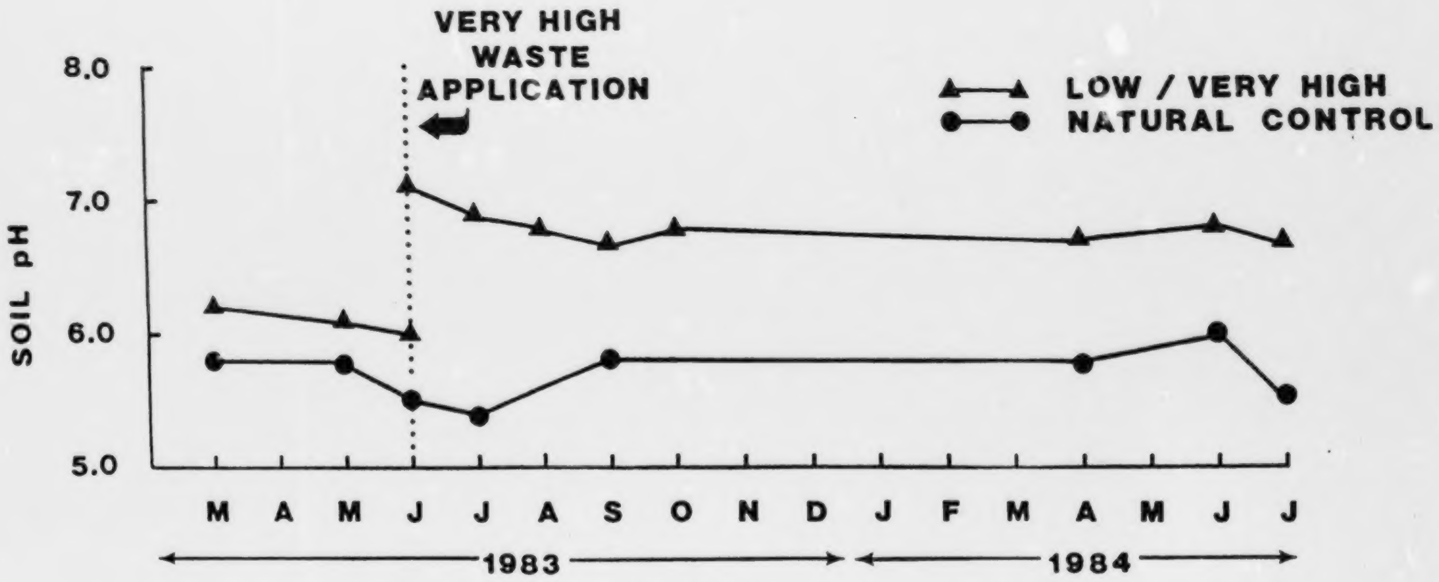


FIGURE 10
EFFECT OF THE JUNE 1983 VERY HIGH WASTE APPLICATION ON THE MEAN VALUE OF SOIL pH, n = 4

Oil and Grease - The oil and grease in the top 15 cm of soil increased as a result of the waste applications (Figure 11). It was not possible to sample the plots immediately after the waste applications. To indicate the loss patterns that occurred, the oil and grease concentrations that should have been in the soil immediately after the June and October 1982 applications were calculated and portrayed on Figure 11. The ranges that occurred in the respective plots are shown in Figure 12 for the very high application rate plots and in Figure 13 for high application rate plots. The range of concentrations were influenced by the spatial variation factors discussed in Appendix C.

With time, the concentration of oil and grease in the soil decreased. However, the applied oil and grease was not lost completely. After each waste application, a new apparent background concentration in the respective plots resulted.

In reviewing the oil and grease losses and accumulations that occurred, it should be recognized that the oily waste came from the bottom of a holding lagoon in Oklahoma. The source of the wastes stored in the lagoon is unknown but probably was from nearby refineries. Typical refinery wastes disposed of in the lagoon may have included oil-water separator sludges, oily tank bottoms, dissolved air flotation (DAF) sludges, and other residual oily materials. While the wastes were stored in the lagoon, some of the volatile compounds may have been lost. Thus the oily wastes applied to the field plots were not necessarily typical of refinery wastes.

The background oil and grease concentration in the soil of the control plots was about 0.4 g oil and grease per gram of moisture free soil. This is comparable to the background data reported for soils at Marcus Hook, Pennsylvania; Tulsa, Oklahoma; and Corpus Christi, Texas (12).

The oil and grease loss pattern had the appearance of a first order type reaction (Figures 11, 12 and 13). The data was analyzed to see if a first order type equation represented the data and if the oil loss could be related to temperature. The resultant correlations, loss rates and calculated half life values are presented in Table 8. Data from the first applications in June 1982 were not used due to the low amounts that were applied and small losses that occurred. The mean values for the respective plots are presented in Appendix F.

As indicated from the correlation coefficients (Table 8), a first order equation was a reasonable assumption for the data. The total oil and grease loss rate constants were essentially the same for the medium, high and very high waste application plots. The loss rate constant for the low waste application plots was higher. The reason for this higher loss rate constant is unknown. It was not related to the type of oil waste applied or to the concentration of oil and grease in the soil. The same waste was applied to all three series of plots (low, medium and high) at the same time (October 1982). The latter plots (medium and high) had

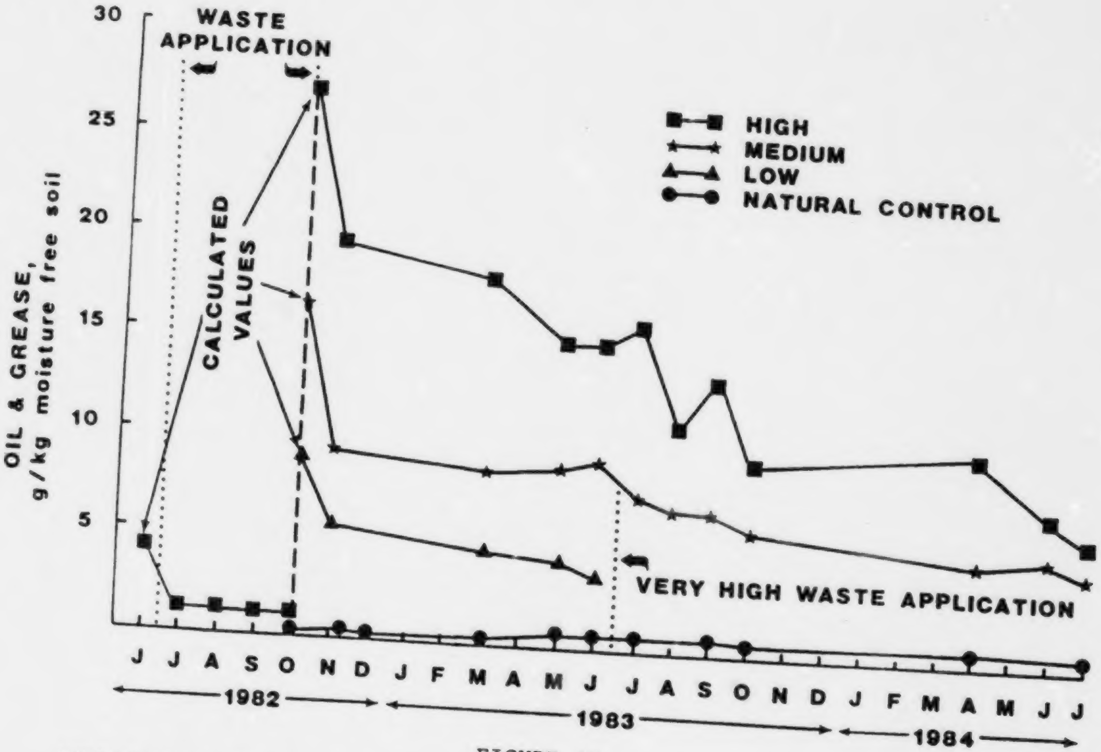


FIGURE 11
AVERAGE OIL AND GREASE CONCENTRATIONS IN THE SOIL OF THE FIELD PLOTS

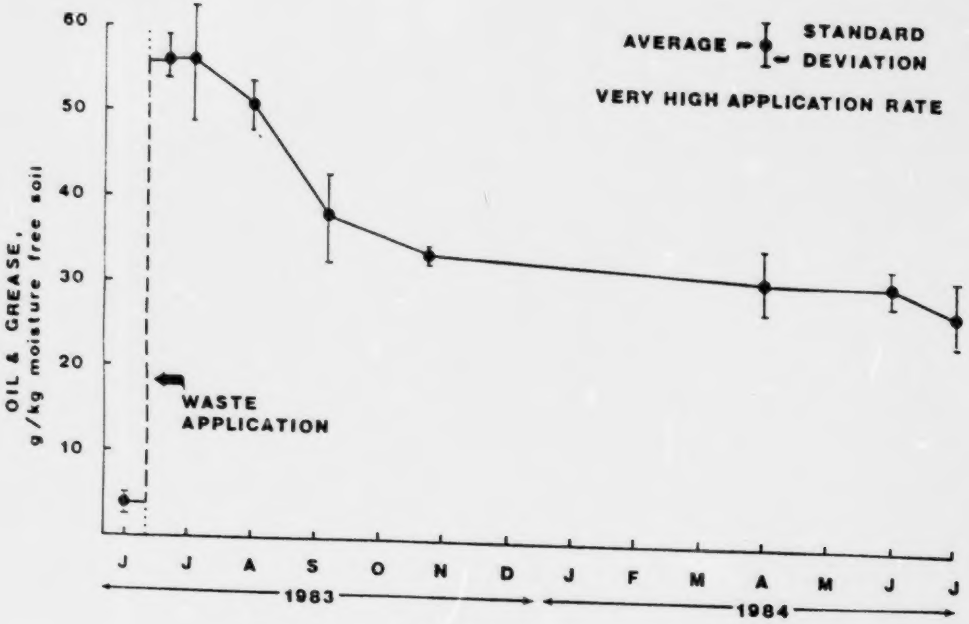


FIGURE 12
AVERAGE OIL AND GREASE CONCENTRATIONS IN THE SOIL OF THE VERY HIGH APPLICATION FIELD PLOTS

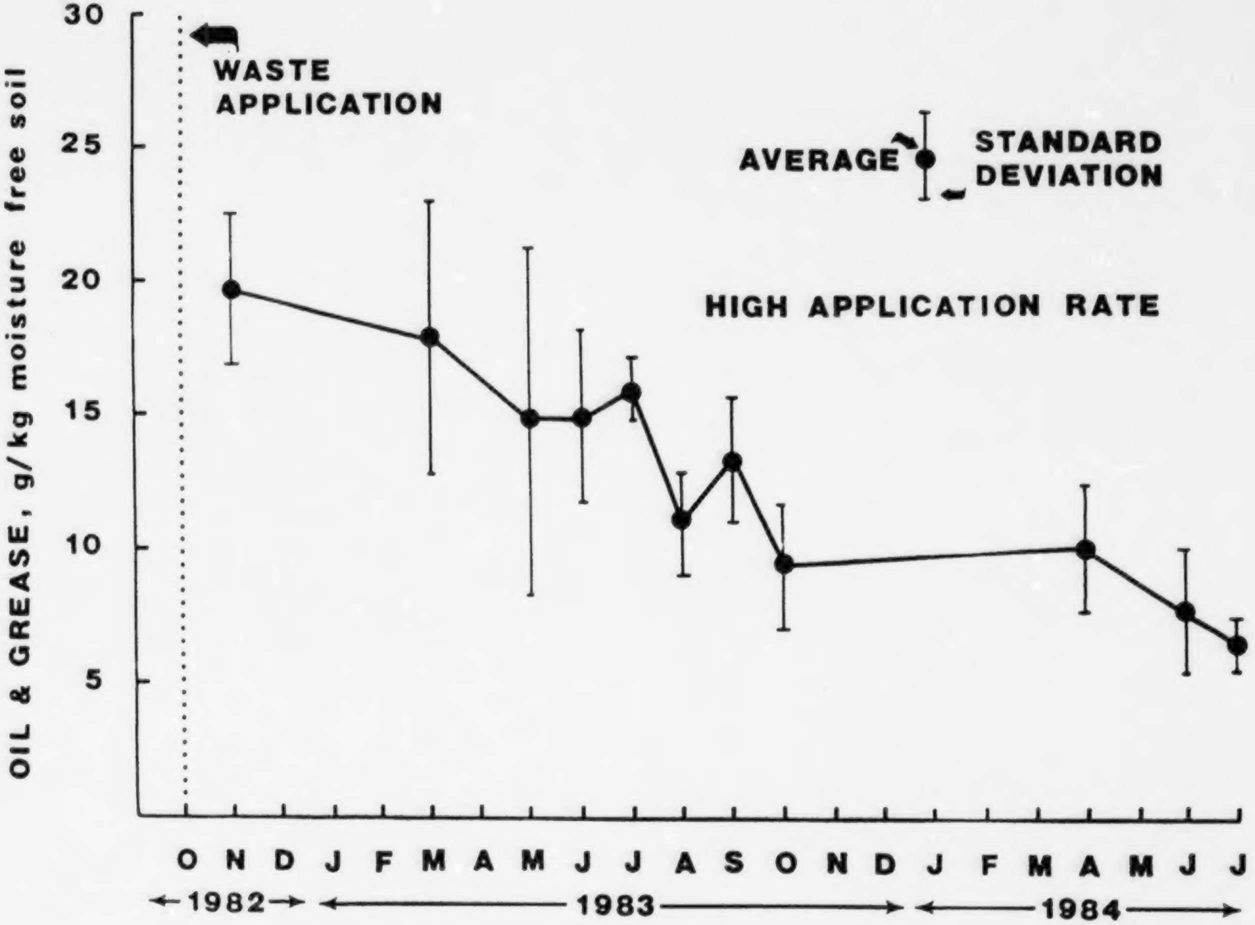


FIGURE 13
AVERAGE OIL AND GREASE CONCENTRATIONS IN THE SOIL OF THE HIGH APPLICATION FIELD PLOTS

TABLE 8. TOTAL OIL AND GREASE LOSS IN THE FIELD PLOTS

Period of Loss	Plots	Loss Rate* Constant (K) ($days^{-1}$)	Correlation Coefficient (R^2)**	Half-Life ($t_{1/2}$)(days)
10/82 to 6/83 (223 days)	Low Application	0.0026	0.83	267
10/82 to 7/84 (612 days)	Medium Application	0.0016	0.88	433
10/82 to 7/84 (612 days)	High Application	0.0018	0.88	385
6/83 to 7/84 (390 days)	Very High Application	0.0017	0.83	408

*The loss rate from a first order equation, i.e. $O_t = O_o e^{-Kt}$ where O_t = total oil and grease concentration in the soil (gram/kg soil MFB) at time t (days), O_o = total oil and grease concentration in the soil at time = zero (t_o), and K = total oil and grease loss rate constant (day^{-1}).

**Correlation coefficient for the first order equation.

higher initial oil and grease concentrations in the soil but had the same loss rate constants.

The loss rates resulted in long half lives ($t_{1/2}$) for the total oil and grease in the respective plots. For the low application plots, the half life was about 300 days while for the medium, high and very high application plots, the half life was about 400 days (Table 8).

It was not possible to statistically correlate the oil and grease losses to the soil temperatures in the field plots. Intuitively, temperature should have an effect on such losses since temperature affects the rates of biodegradation and of volatilization, the most likely mechanisms of loss in the field plots. However, any effect due to temperature could not be discerned and separated from other parameters affecting the oil losses. The effect of temperature probably was masked by factors such as the variability in the oil and grease data, differences in soil moisture as the soil temperature changed, and the different oil and grease compounds that were present in the soil at different times during the study. The oil and grease loss patterns (Figures 11, 12 and 13) do indicate greater losses during the warmer periods of a year and less loss during the colder periods.

Although a first order equation apparently was a reasonable mathematical expression to portray the oil and grease losses, realistically it is not completely acceptable since first order equations of the type shown in Table 8 indicate that eventually there will be a complete loss. The data, however, suggests otherwise. Some of the applied oil and grease appeared to remain in the soil even after twelve or more months.

The oil and grease losses had not completely ceased by the last sampling date in July 1984. The available data was analyzed to determine the maximum amount of loss that would occur and, by difference, to determine the "biodegradable" fraction of the applied waste. The results are presented in Table 9. There is the possibility that the waste applied in June 1983 was less degradable than the waste applied in October 1982 even though: (a) the general characteristics of the applied wastes (Table 1) were not drastically different on a moisture free basis, and (b) and the total oil and grease loss rates for the different wastes were essentially the same (Table 8).

The constituents of the refractory fraction of the wastes were not determined. It is postulated that they are long chain, high molecular weight oily compounds such as asphaltenes, paraffins and similar compounds.

Even though there was an apparent large accumulation of oil and grease that resulted from the very high application in June 1983, these ultimate residuals may not have any adverse environmental impact. As was noted earlier (SECTION 4), soil from the very high plots was used in the artificial soil test to determine the impact of the residuals to earthworms. The results of these tests (Table 6) indicated that any adverse impact decreased with time. As the artificial soil test results note, the

TABLE 9. MAXIMUM ESTIMATED OIL AND GREASE LOSS IN THE FIELD PLOTS

Plots	Maximum Estimated Loss*	Refractory Fraction**
Waste Applied October 1982		
Low Application	78%	22%
Medium Application	76%	24%
High Application	80%	20%
Waste Applied June 1983		
Very High Application	52%+	48%

*The loss that would occur over a period of time longer than that of the study. This loss is equivalent to the fraction of the applied waste that is able to be lost by biodegradation, volatilization or other mechanisms.

**The fraction of applied material estimated to remain in the soil from the noted application.

+The maximum predicted loss for the very high application was obtained using data collected over 390 days whereas the maximum predicted losses for the low, medium and high applications were obtained using data collected over 612 days. When data collected over the first 350 days after the waste was applied was used to estimate the maximum loss, the maximum predicted loss for the low, medium, and high application plots was about 40% whereas for the very high application plots, it was 52%. It is possible that a smaller refractory fraction would have estimated from the very high application data had there been time to collect more loss data over a longer time period (such as over 600 days).

residuals from the medium and high applications of October 1982 had practically no effect on the earthworms. The inference that the refractory residuals may not have an adverse impact also is verified by data on earthworm numbers and biomass discussed later in this SECTION.

Temperature - The soil temperature in the field plots are portrayed in Figure 14. The temperatures of the plots receiving the oily waste did not increase after the first waste application in June 1982. Because of the cold weather after the October 1983 waste application, there also was no difference in soil temperature of all the plots until the summer of 1983. During that summer, the soil temperature of the plots receiving the high waste application was greater than that of the other plots by 1 to 1.5°C. During the period of June 1982 through May 1983, the temperature variation among the means for the different plots was less than 1°C. The temperatures of the high application rate plots were no different than that of the natural control plots by the fall of 1983.

After the very high waste application was made to plots 5, 6, 12 and 18 in June 1983, the soil temperature of these plots was noticeably higher than that of the other plots (Figure 14). The temperature of the incorporation zone of the very high plots was from 3 to 5°C greater than that of the natural controls. The temperature increase may be due to the absence of surface vegetation and the darker color of the surface soil.

Although the temperatures in the very high plots decreased to that of the natural controls by November 1983, the increase again occurred during the spring and summer of 1984 (Figure 15). During this period, no tilling occurred, no vegetation grew on the very high plots and the color of the soil continued to be darker than that of the other plots.

Soil Moisture - The moisture content of the top 15 cm of the soil in the field plots changed throughout the project (Figure 16), decreasing during the summer months. Generally the soil moisture ranged from 20 to 32% on a wet basis.

The soil moisture pattern was the reverse of that of soil temperature (Figure 8) which increased during the summer months. Both soil moisture and soil temperature will have an effect on the soil biota.

Cation Exchange Capacity - The cation exchange capacity (CEC) of the soils in the field plots was analyzed periodically throughout the project. The purpose of the analyses was to ascertain whether the application of the oily waste had any effect on the CEC of the soil. There were some variations between the plots but no trend with time or with the waste application rate was identified. The average CEC values for the specific types of plots are noted in Table 10. The detailed CEC data are summarized in Appendix E.

Metals - The average metal concentrations in the soil at the field plots are summarized in Appendix G. Although there were high concentrations of certain metals in the wastes (Table 2), the application of the

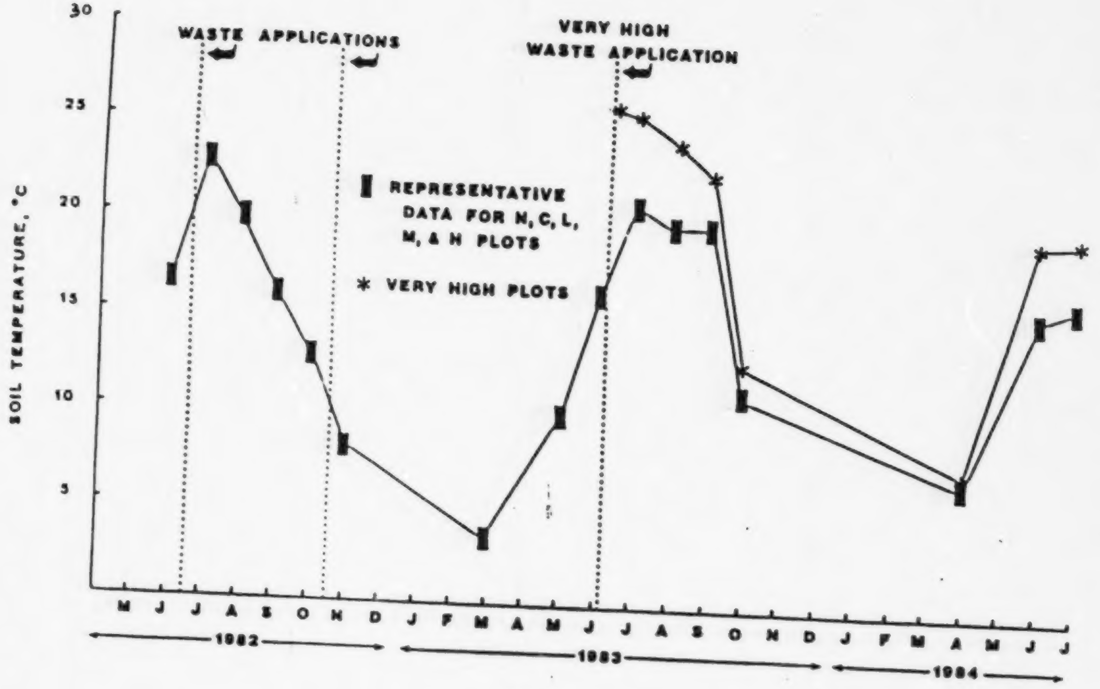


FIGURE 14
TEMPERATURE OF SOIL IN THE FIELD PLOTS
DURING THE STUDY

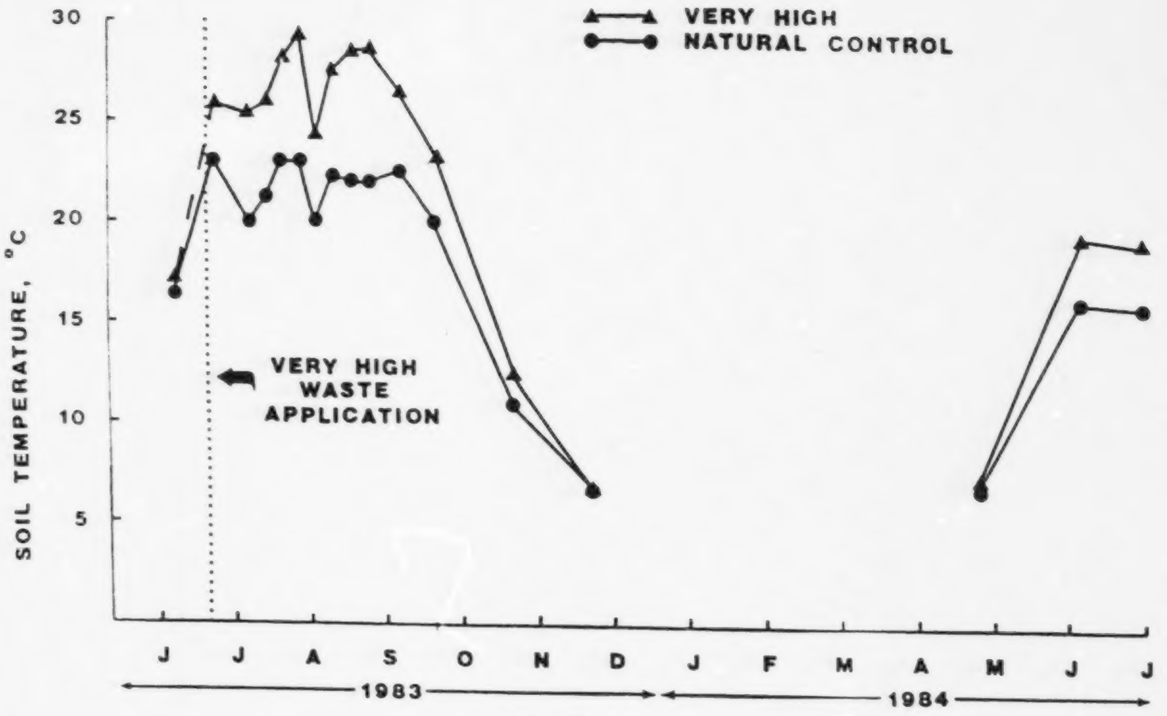


FIGURE 15
SOIL TEMPERATURES IN THE NATURAL CONTROL AND VERY HIGH APPLICATION PLOTS - 1983 - 1984

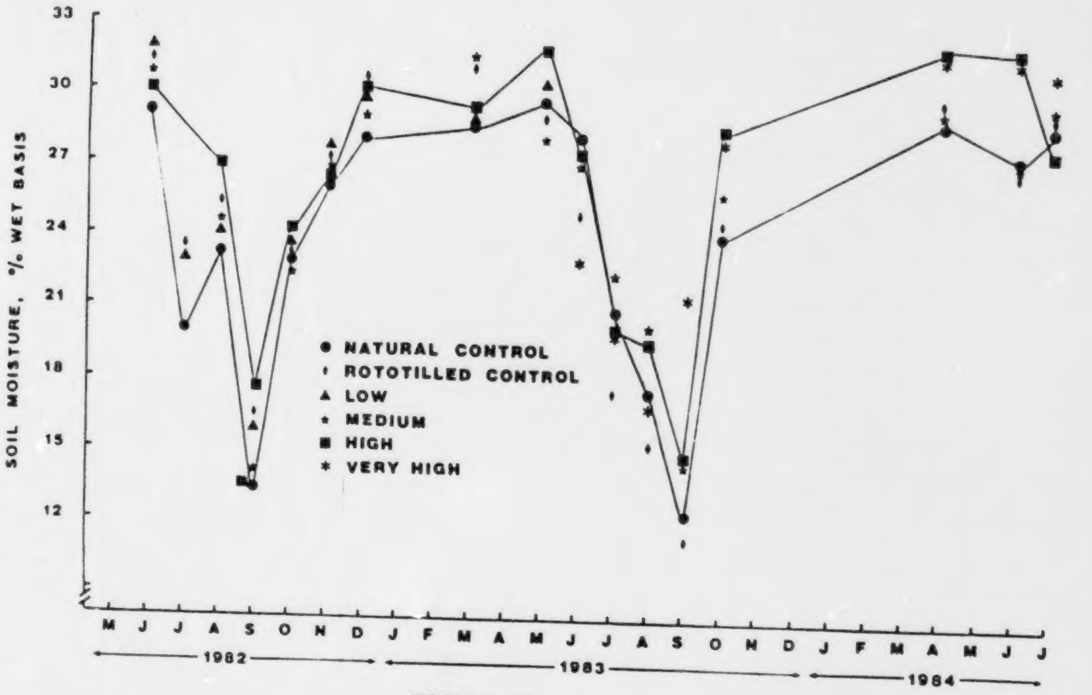


FIGURE 16
AVERAGE SOIL MOISTURE IN THE FIELD PLOTS - 1982 - 1984

wastes did not always result in measurable increases in metal concentrations in the soil. This was particularly true for the applications in June 1982 and for some of the applications in October 1983.

TABLE 10. AVERAGE CEC VALUES FOR THE SOILS AT THE FIELD SITE

Type of Application	CEC Values (meq/100 gram)*
Natural Control	24.8 ± 4.6
Rototilled Control	26.0 ± 5.8
Low	25.5 ± 3.8
Medium	24.5 ± 3.2
High	25.9 ± 5.0
Very High	24.0 ± 4.4

*Average and standard deviation.

Mass balance estimates were made to identify the increase in soil metal concentrations that should have occurred as a result of the seven waste applications. This was done by calculating: (a) the metal concentration in the top 15 cm of the soil before an application, and (b) the metal concentration in the top 15 cm that should have resulted after the waste was applied and rototilled into the top 15 cm. These independent calculations were done to ascertain whether the waste applications should have resulted in a measurable increase in the soil metal concentrations. Such calculations also avoided any differences that may have been caused by incomplete rototilling and spatial variations in the samples taken after the waste application. The metal concentrations calculated to be in the soils after the waste applications were consistent with the analysed metal concentrations in the soil samples taken after the waste applications.

Table 11 summarizes the increases of the soil metal concentration that were calculated to result from each waste application. The percentage values in the table represent the increase over the concentration of the metal in the soil immediately before the waste application. For example, the June 1983 waste application to the very high plots increased aluminum concentration 3.6% over what the concentration was on the soil sampling data immediately before the June 1983 application.

The June 1982 waste applications did not increase the soil metal concentrations measurably, except perhaps the calcium, chromium, sodium and zinc concentrations following the high waste application of that date. The October 1982 waste applications were greater and the soil metal concentrations of these four metals and of copper and lead also were

TABLE 11. PERCENT INCREASE* IN SOIL METAL CONCENTRATIONS AS A RESULT OF THE WASTE APPLICATIONS

	Date of Waste Application						
	June 1982			October 1982			June 1983
Metal	Low	Med.	High	Low	Med.	High	Very High
Al	0.03	0.06	0.1	0.5	1	1.5	3.6
Ca	2	5	9	19	39	56	174
Cd	0.5	1	2	5	8	13	51
Cr	3	5	10	20	48	68	160
Cu	1	2	4	8	15	24	93
Fe	0.03	0.05	0.09	0.3	0.6	0.1	3.5
K	0.06	0.09	0.2	1	2.4	3.6	5.3
Mg	0.05	0.09	0.2	0.6	1.2	1.8	5.1
Mn	0.02	0.04	0.07	0.2	0.5	0.7	2.8
Na	1	2	5	5	10	16	95
Ni	0.06	0.1	0.2	0.8	1.7	2.6	9
Pb	0.7	1	3	8	15	24	99
Ti	0.08	0.1	0.3	2	3	4	12
V	0.03	0.06	0.1	0.5	1	1.5	3.8
Zn	2	4	8	20	34	50	133

*increase over the concentration of the metal in the soil immediately before the waste application; values were calculated using the quantity of waste applied, the metal concentration of the waste, and the metal concentration in the soil before the application.

TABLE 12. STATISTICAL ANALYSIS* OF THE CHANGES** IN SOIL METAL CONCENTRATIONS (0-15 cm) AFTER THE WASTE APPLICATIONS

	Difference After Application of June 1982			Difference After Application of October 1982			Difference After Application of June 1983
Metal	Low	Med.	High	Low	Med.	High	Very High
Al	0	0	0	0	0	0	0
Ca	0	0	0	0	+	+	+
Cd	0	0	0	0	0	0	+
Cr	0	0	+	+	+	+	+
Cu	0	0	0	0	0	+	+
Fe	0	0	0	0	0	0	0
K	0	0	0	0	0	0	+
Mg	0	0	0	0	0	0	0
Mn	0	0	0	0	0	0	0
Na	0	0	0	0	0	0	+
Ni	0	0	0	0	0	0	0
Pb	0	0	0	+	+	+	+
Ti	0	0	0	0	0	0	+
V	0	0	0	0	0	0	0
Zn	0	0	0	+	+	+	+

* 0 = no significant change ($P \leq 0.05$) as a result of the waste application

+ = a significant increase ($P \leq 0.05$) as a result of the waste application

** change over the concentration of the metal in the soil immediately before the waste application increased measurably. The very high application of June 1983 increased the soil metal concentrations of many of the metals.

The differences also were statistically analyzed to determine when significant increases occurred. The analyses were a one way ANOVA followed by Duncan's new multiple range test when significance was found. The comparison is presented in Table 12 and clearly indicates when the soil metal concentrations increased significantly.

The increases in calcium undoubtedly resulted in the pH increase of the high and very high application plots (Figures 9 and 10). Increases in soil pH increase the immobilization of most metals in the soil.

The increases for chromium, copper and zinc are portrayed in Figures 17 and 18. The soil concentrations of nickel also are presented in Figure 18 to illustrate the type of variations that occurred for a metal that did not increase in the soil.

The data for several metals (chromium, copper, lead and zinc) that are of potential environmental concern were evaluated to determine if the metal concentrations increased over time as a result of the waste applications. The statistical analysis indicated that the metal concentrations in the upper 15 cm of the soil did not decrease with time. Example results are presented in Table 13 for chromium and zinc and in Table 14 for the copper and lead soil concentrations. The data show no change in the metal concentration for all metals in the natural and rototilled controls over the entire study.

The immobilization of metals in the soil was analyzed by comparing the metal concentrations of subsoil samples from the 15 to 30 cm depth taken in October 1983. The metal concentrations of subsoil samples from the plots to which the wastes were applied were analyzed statistically to determine if the deeper soils of the controls and the waste application plots had different metal concentrations. As of the October 1983 sampling date, the wastes had been applied to the medium and high application plots for about one year and had been applied to the very high application plots for about four months.

The statistical analysis (Table 15) indicated that sodium was the only metal that had a significantly different concentration in the 15 to 30 cm depth between the control plots and any waste application plot. That difference only occurred for sodium in the soil of the very high plots. Sodium was in high concentrations in the applied waste (Table 2) and can be a mobile ion under certain conditions. Thus, all of the other metals were immobilized in the top 15 cm of the plots.

Organics - Soil samples were extracted with methylene chloride and the extracts analyzed for organics (SECTION 3). Due to time and personnel constraints, it was impossible to analyze for the organic compounds found in the oily waste (Table 3). Rather a smaller number of organic compounds were analyzed in selected soil samples in order to determine the loss of these compounds after application.

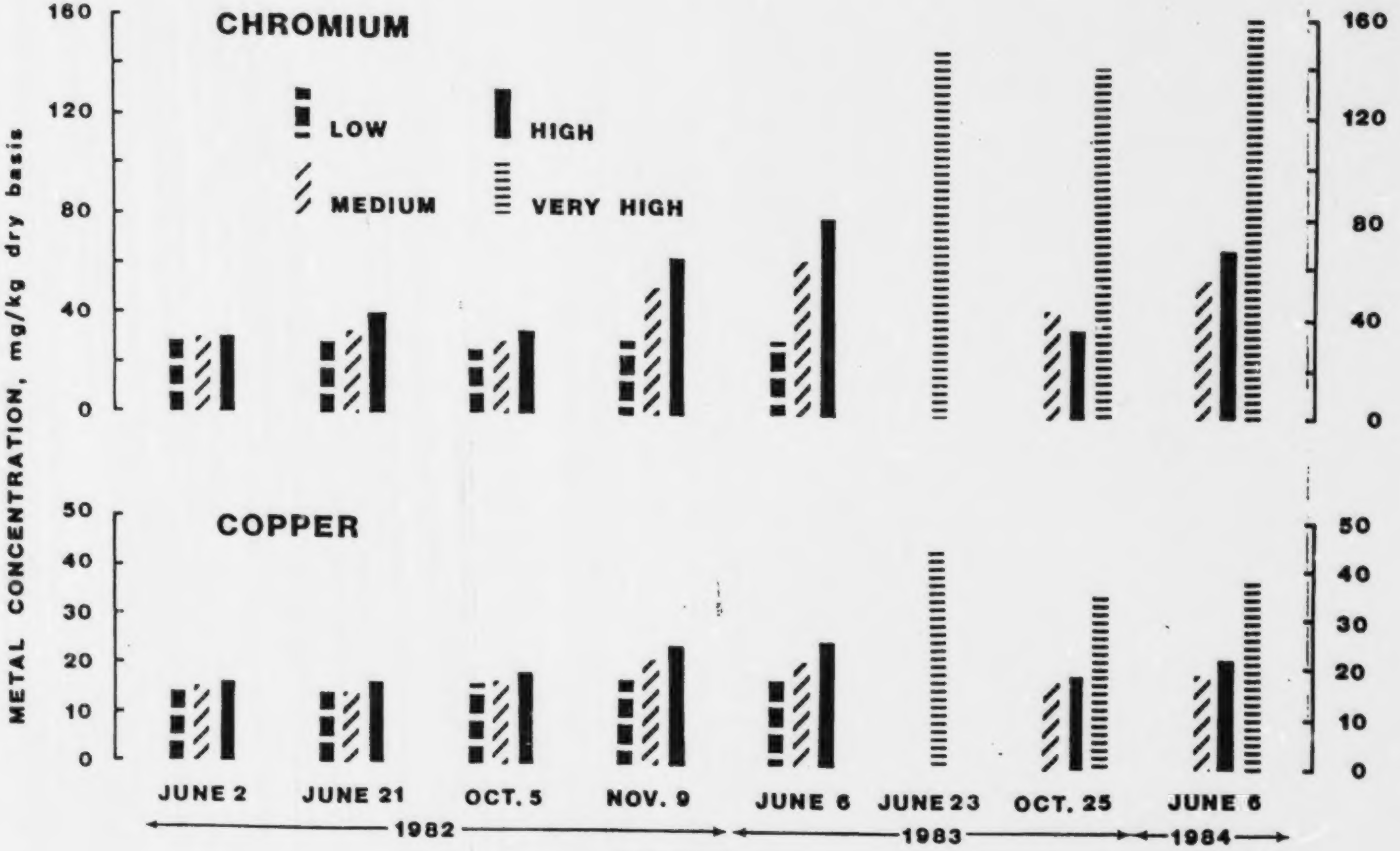


FIGURE 17
CHROMIUM AND COPPER CONCENTRATIONS IN THE SOILS OF THE FIELD PLOTS

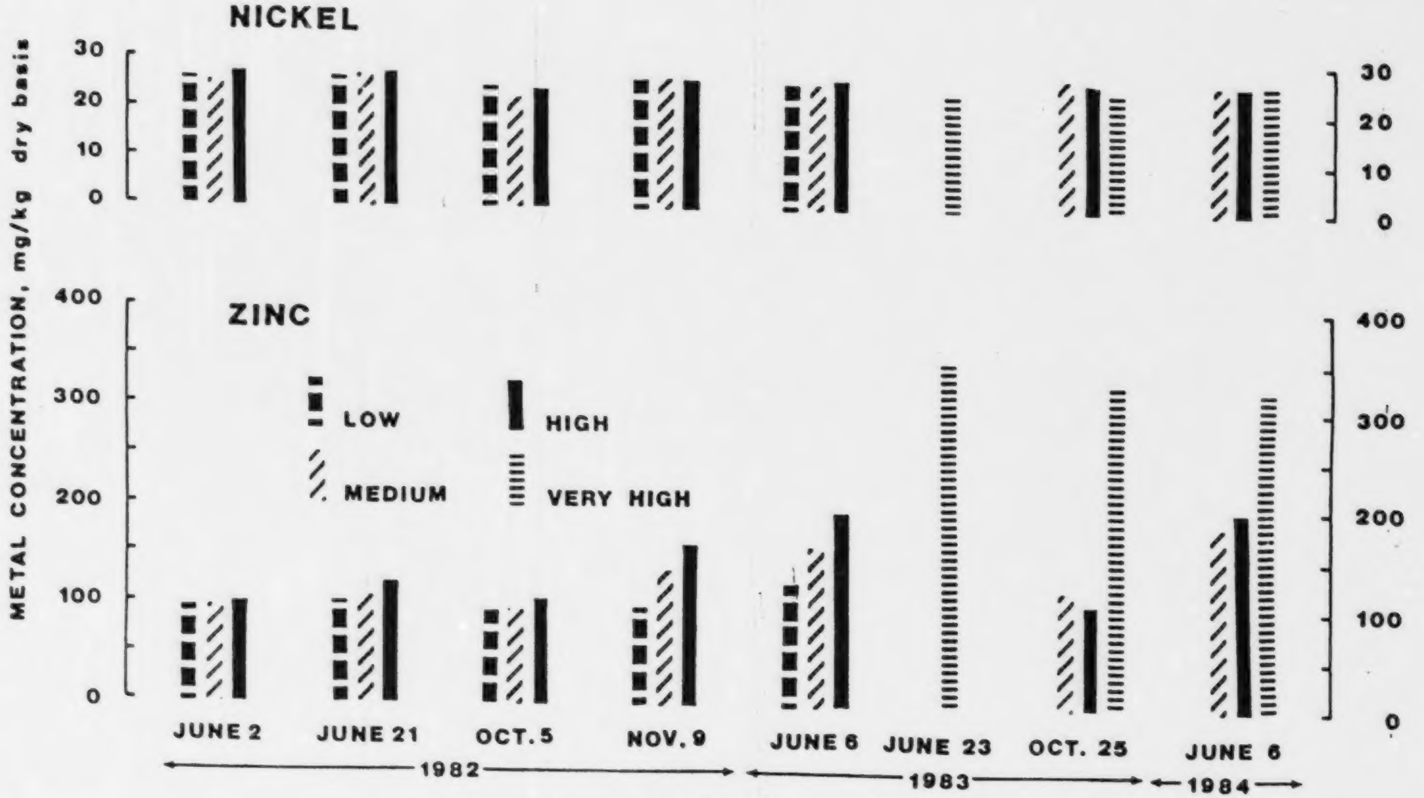


FIGURE 18
NICKEL AND ZINC CONCENTRATIONS IN THE SOILS OF THE FIELD PLOTS

TABLE 13. AVERAGE CHROMIUM AND ZINC CONCENTRATIONS^{+} IN THE TOP 15 CENTIMETERS OF THE SOIL OF THE FIELD PLOTS — STATISTICAL ANALYSIS*

Plots	Date of Sample^{++}							
	6/2/82	6/21/82	10/5/82	11/9/82	6/6/83	6/21/83	10/25/83	6/6/84
Chromium								
Natural Control	31^{a}	29^{a}	27^{a}	29^{a}	30^{a}	--	27^{a}	30^{a}
Rototilled Control	31^{a}	30^{a}	27^{a}	25^{a}	31^{a}	--	27^{a}	29^{a}
Low Application	29^{a}	32^{a}	29^{a}	48^{b}	41^{b}	--	--	--
Medium Application	30^{a}	33^{a}	29^{a}	51^{b}	62^{b}	--	43^{a}	80^{b}
High Application	31^{a}	39^{a}	33^{a}	63^{b}	83^{c}	--	36^{a}	68^{b}
Very High Application**	--	--	--	--	--	145^{a}	139^{a}	136^{a}
Zinc								
Natural Control	98^{a}	99^{a}	93^{a}	99^{a}	98^{a}	--	92^{a}	106^{a}
Rototilled Control	98^{a}	98^{a}	93^{a}	96^{a}	98^{a}	--	88^{a}	100^{a}
Low Application	101^{a}	105^{a}	101^{a}	135^{a}	114^{a}	--	--	--
Medium Application	97^{a}	104^{a}	95^{a}	136^{b}	160^{b}	--	120^{a}	203^{b}
High Application	101^{a}	120^{a}	106^{a}	163^{b}	200^{b}	--	107^{a}	184^{b}
Very High Application	--	--	--	--	--	345^{a}	325^{a}	331^{a}

+ mg/kg soil MFB, n=4.

* data with the same superscript in a horizontal row are not statistically different at the 95% confidence level ($P<0.05$).

++ waste applications were in mid-June 1982, late October 1982 and mid-June 1983.

** In June 1983, the low application plots became the very high application plots (see text SECTION 5).

TABLE 14. AVERAGE COPPER AND LEAD CONCENTRATIONS+ IN THE TOP 15 CENTIMETERS OF THE SOIL OF THE FIELD PLOTS -- STATISTICAL ANALYSIS*

Plots	Date of Sample++							
	6/2/82	6/21/82	10/5/82	11/9/82	6/6/83	6/21/83	10/25/83	6/6/84
Copper								
Natural Control	14^a	14^a	16^a	17^a	16^a	--	16^a	20^a
Rototilled Control	15^a	15^a	18^a	20^a	18^a	--	15^a	21^a
Low Application	14^a	15^a	17^a	21^a	19^a	--	--	--
Medium Application	15^a	15^a	17^a	21^b	21^b	--	17^a	21^b
High Application	16^a	16^a	18^a	24^b	25^b	--	19^a	22^{ab}
Very High Application**	--	--	--	--	--	43^b	35^b	37^b
Lead								
Natural Control	26^a	26^a	24^a	25^a	25^a	--	19^b	19^b
Rototilled Control	25^a	27^a	23^{ab}	25^a	24^a	--	20^{ab}	19^b
Low Application	26^a	27^a	23^b	31^a	29^a	--	--	--
Medium Application	26^a	27^{ab}	24^a	32^b	30^{ab}	--	22^a	24^a
High Application	25^a	30^{ab}	25^a	35^b	34^b	--	22^a	27^a
Very High Application	--	--	--	--	--	53^b	48^b	49^b

+ mg/kg soil MFB, n=4.

* data with the same superscript in a horizontal row are not statistically different at the 95% confidence level ($P<0.05$).

++ waste applications were in mid-June 1982, late October 1982 and mid-June 1983.

** In June 1983, the low application plots became the very high application plots (see text SECTION 5).

TABLE 15. METAL CONCENTRATIONS$^+$ IN SUBSURFACE SOILS (15 TO 30 cm DEPTH) AT THE FIELD PLOTS

Metal	Natural Control	Rototilled Control	Medium Application	High Application	Very High Application	F Value
Aluminum	24,400 ±1,100	25,200 ±2,600	23,500 ±1,300	25,500 ±1,600	27,300 ±1,500	2.11
Cadmium	<*	<	<	<	<	-
Calcium	2,900 ±900	3,400 ±1,300	3,000 ±800	3,500 ±500	3,600 ±1,400	0.24
Chromium	28 ±2	29 ±2	27 ±2	30 ±2	38 ±10	2.23
Copper	15 ±3	16 ±3	14 ±2	16 ±2	17 ±2	0.36
Iron	28,300 ±2,400	28,600 ±2,200	27,700 ±2,500	28,300 ±1,600	28,800 ±1,700	0.12
Lead	18 ±3	18 ±3	20 ±4	17 ±2	19 ±3	0.38
Magnesium	4,600 ±200	4,600 ±200	4,400 ±200	4,600 ±200	4,800 ±100	1.35
Manganese	560 ±190	670 ±230	540 ±130	730 ±210	650 ±140	0.56
Nickel	26 ±2	26 ±2	25 ±2	27 ±2	26 ±1	0.46

(continued)

TABLE 15. (continued)

Potassium	2,400 ±300	2,500 ±500	2,200 ±500	2,400 ±400	3,200 ±700	1.85
Sodium	84^{a} ±3	84^{a} ±13	90^{a} ±27	105^{a} ±14	144^{b} ±32	4.47**
Titanium	74 ±19	65 ±14	60 ±26	70 ±7	109 ±38	2.05
Vanadium	38 ±2	38 ±4	36 ±3	39 ±3	42 ±3	1.89
Zinc	86 ±7	85 ±12	82 ±6	90 ±13	100 ±23	0.77

+sampled on October 15, 1983; average and standard deviation, n=4.

*less than limit of detection determined by instrument sensitivity, sample dilution and analytical matrix interference.

**statistically significant difference ($P \leq 0.05$) as a result of the very high application data; only the results for sodium showed any significant differences.

Emphasis was placed on soil samples from the very high application plots since it was expected that the concentration of organic compounds in these plots would be well above detection limits and might remain so for a reasonable period of time. Soil samples from such plots (plots 5 and 12) were taken shortly after the application in June 1983 and monthly thereafter through October 1983. In addition, soil samples from plot 18 (a very high plot), plot 14 (a high plot) and plot 11 (a medium plot) were analyzed at longer time intervals to either confirm the loss patterns from plots 5 and 12 or to identify the losses in the plots that had received lower waste applications.

The organic compounds that were determined in the extracts included C_8 to C_{26} alkanes, napthalenes and several other aromatics such as fluorene, anthracene, phenanthrene and pyrene. The concentration of these organics that were found in the soil samples are summarized in Appendix H.

Immediately after the very high waste application, almost all of the noted organic compounds were present in the soil samples above detection limits. The organics in the highest concentrations included napthalene and several methyl-napthalenes, C_{10} thru C_{26} alkanes, and a number of other aromatics. Less than one month after the very high waste application, considerable loss of most of the organics had occurred. Only the C_{14} to C_{25} alkanes could be detected several months after the waste application. The chromatograms shown in Figures 19 and 20 illustrate the changes in organic compounds that occurred.

The data for the high application plot (plot 14) and the medium application plot (plot 11) indicated the same general pattern as that for the very high application plots. The C_{15} to C_{26} alkanes were present in the highest concentration. One difference was that no napthalenes or other aromatics were detected in the soil samples shortly after the high and medium waste applications. Another noticeable difference is that C_{16} to C_{23} alkanes were present at low but detectable concentrations in the soil of the high application plot (plot 14) in June 1983, over seven months after the wastes were applied. These seven months covered the period of November through May, the colder time of the year. In contrast, none of the organics could be detected in the soil samples of the very high plots four months after the very high waste application. These four months covered the period of June through October, the warmest months of the year.

Because many of the organic compounds were lost rapidly from the plots, it was not possible to estimate their loss rates. However, where two or more concentrations of an organic compound were above the detection limits, a first order equation was assumed to fit the data and the loss rates were estimated. Table 16 summarizes the estimated loss rates.

The loss rate constants for the specific organic compounds indicate that during the warmer months (June through October), the losses were rapid with half-lives generally less than 30 days. The apparent longer half-life data for plots 18 and 14 should be considered cautiously since only two data points were used to calculate the loss rate constants and

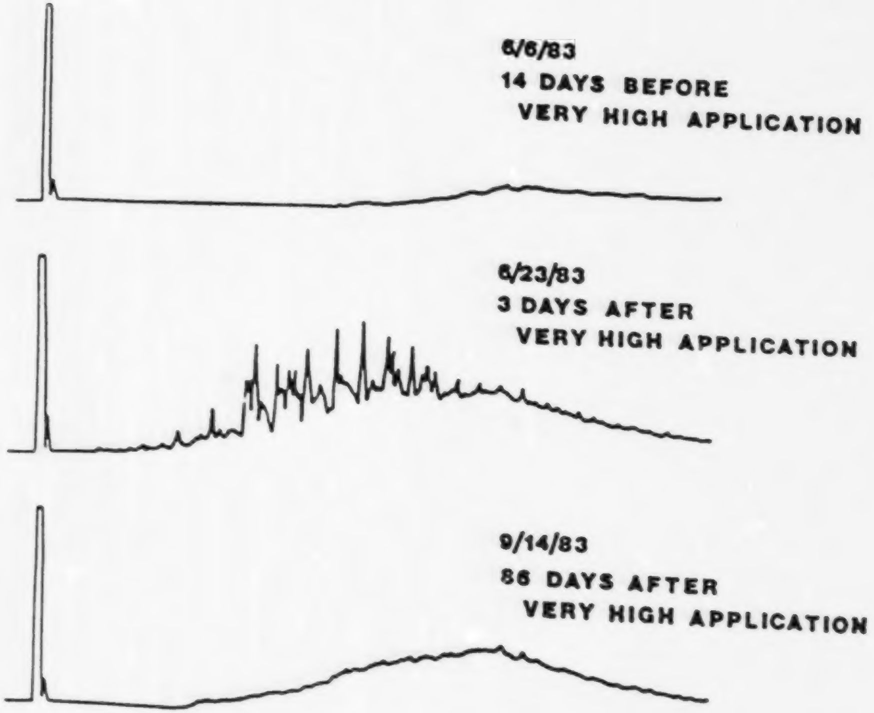


FIGURE 19
CHROMATOGRAMS OF ORGANICS EXTRACTED FROM THE SOIL OF PLOT 5
A VERY HIGH APPLICATION PLOT

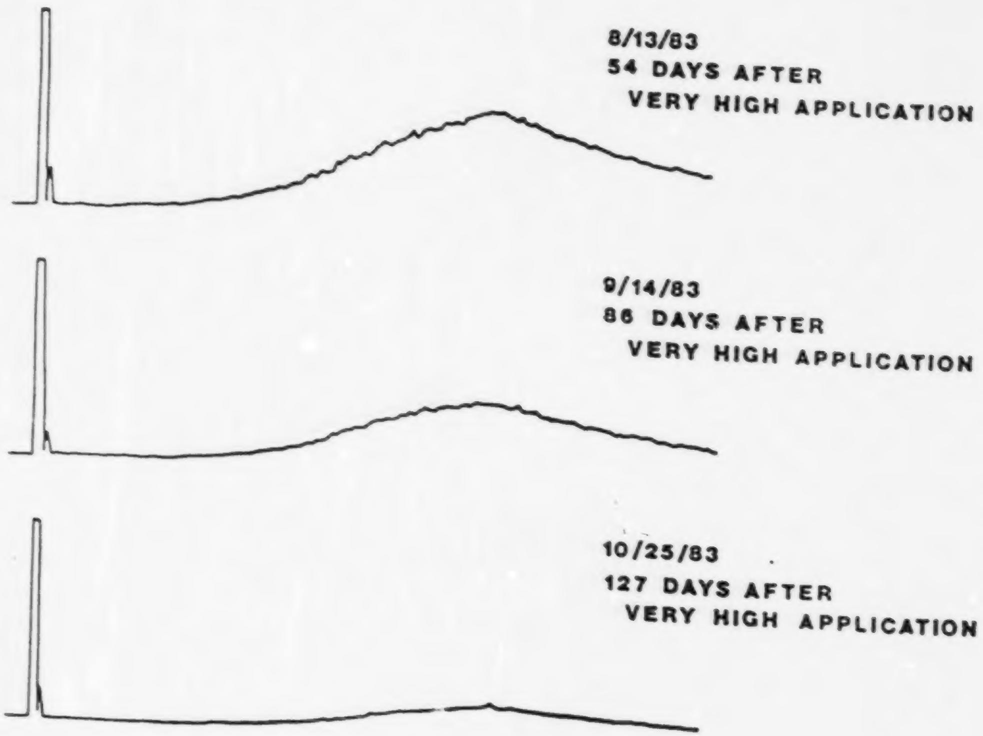


FIGURE 20
CHROMATOGRAMS OF ORGANICS EXTRACTED FROM THE SOIL OF PLOT 12
A VERY HIGH APPLICATION PLOT

TABLE 16. ESTIMATED LOSS OF ORGANIC COMPOUNDS IN THE FIELD PLOTS+

Organic Compound	Plot 5		Plot 12		Plot 18		Plot 14	
	Loss Rate Constant (K,day^{-1})*	Calculated Half-Life (days)	Loss Rate Constant (K,day^{-1})	Calculated Half-Life (days)	Loss Rate Constant (K,day^{-1})++	Calculated Half-Life (days)	Loss Rate Constant (K,day^{-1})++	Calculated Half-Life (days)
Napthalene	R**	-	R	-	R	-	-	-
2-methyl-napthalene	0.048	14	0.03	23	R	-	-	-
1-methyl-napthalene	0.056	12	R	-	R	-	-	-
2,6-dimethyl-napthalene	0.094	7	R	-	R	-	-	-
1,3-dimethyl-napthalene	0.10	7	0.08	9	R	-	-	-
2,3-dimethyl-napthalene	0.08	9	0.06	11	R	-	-	-
1,2-dimethyl-napthalene	0.07	10	R	-	R	-	-	-
C_8 Alkane	-	-	-	-	-	-	-	-
C_9 Alkane	-	-	-	-	-	-	-	-
C_{10} Alkane	R	-	R	-	-	-	-	-
C_{11} Alkane	R	-	R	-	-	-	-	-
C_{12} Alkane	0.06	11	0.05	15	-	-	-	-
C_{13} Alkane	0.07	10	0.07	10	R	-	-	-
C_{14} Alkane	0.085	8	0.04	18	-	-	-	-
C_{15} Alkane	0.028	25	0.03	23	0.005	138	-	-
C_{16} Alkane	0.095	7	0.023	30	R	-	0.005	138
C_{17} Alkane	0.03	23	0.024	29	0.005	138	-	-
C_{18} Alkane	0.03	23	0.026	27	0.006	115	0.006	115
C_{19} Alkane	0.03	23	0.026	27	0.006	115	0.006	115
C_{20} Alkane	0.03	23	0.025	28	0.006	116	0.006	115
C_{21} Alkane	0.078	9	0.022	32	-	-	-	-
C_{22} Alkane	0.067	10	0.03	23	-	-	0.003	230
C_{23} Alkane	0.06	11	0.026	27	0.003	230	-	-
C_{24} Alkane	0.06	11	0.026	27	-	-	-	-
C_{25} Alkane	0.076	9	0.015	45	-	-	-	-
C_{26} Alkane	0.05	13	0.02	34	0.005	138	-	-
Biphenyl	R	-	0.03	23	-	-	-	-
3-methylbiphenyl	R	-	0.035	20	R	-	-	-
Dibenzofuran	R	-	0.04	17	R	-	-	-
Fluorene	R	-	0.02	35	-	-	-	-

TABLE 16. (continued)

Phenanthrene	R	-	R	-	-	-	-	-
Anthracene	R	-	R	-	-	-	-	-
Carbazole	-	-	-	-	-	-	-	-
1-Methylphenanthrene	R	-	0.03	23	R	-	-	-
Pyrene	0.013	54	0.007	99	-	-	-	-

[+] Plots 5, 12 and 18 are very high application plots, Plot 14 is a high application plot.

[++] Only two data points were available to calculate these loss rates and the concentrations of the second set of data were very close to the detectable limits.

[*] The loss rate from a first order equation, i.e. $O_t = O_o e^{-Kt}$ where O_t and O_o = concentration in the soil (mg/kg MFB) at time t and at time = zero, and K = loss rate constant (day^{-1}).

[**] Rapid - loss to below detectable limits occurred in less than one month after application.

the concentrations of the second data point were very close to the detection limits of the analytical methods that were used. In contrast, in plots 5 and 12, there were at least three and as many as five data points for many of the organic compounds and almost all of the concentrations were considerably above the detection limits (Appendix H).

Because of the limited data, it was not possible to relate the loss rate constants to the soil temperature or other factors that might affect the loss of the organic compounds. The fact that some of the alkanes were able to be detected in the high application plot (plot 14) after seven months of cold weather suggests that the loss rates were lower during the winter months. The loss rates were rapid during the summer months following the very high application in June 1983.

The losses for these organic compounds (Table 16) occurred much more rapidly than did the loss of total oil and grease (Table 8). Whereas the half-life of these organic compounds was generally less than 30 days, the half-life of the total oil and grease ranged from 260 to over 400 days. The difference is caused by other organic compounds that are not lost as rapidly as the identified compounds but are measured by the oil and grease analysis.

As discussed earlier, only certain organic compounds were able to be determined quantitatively in the methylene chloride extracted soil samples. Although most of these compounds were lost in a short period of time (several months), other organic compounds remained in the soil for much longer periods of time (Figures 11, 12, and 13 and Table 9). The GC/MS preparatory procedures used by RSKERL found considerable quantities of such residual organics in the extracts. The separation and identification procedures suggested the branched alkanes were among such residual organics. No other specific organics could be adequately separated from the residual organics and identified.

EARTHWORM DATA

Biomass and Numbers

The average biomass and numbers of earthworms found in the zone of incorporation of the field plots are summarized in Appendix I. The patterns of earthworm biomass and numbers that occurred throughout the study are presented in Figures 21 and 22 respectively.

Allolobophora and *Lumbricus* were the genera of worms that predominated in the field plots. These are worms commonly found under field conditions in the eastern United States. They are most commonly found in the upper soils in the spring and fall when moisture and temperature conditions are more favorable. In reporting the earthworm data, the biomass data and the numbers of both genera have been combined.

A one-way analysis of variance was performed on the data for each sampling period to test the null hypothesis that there was no difference

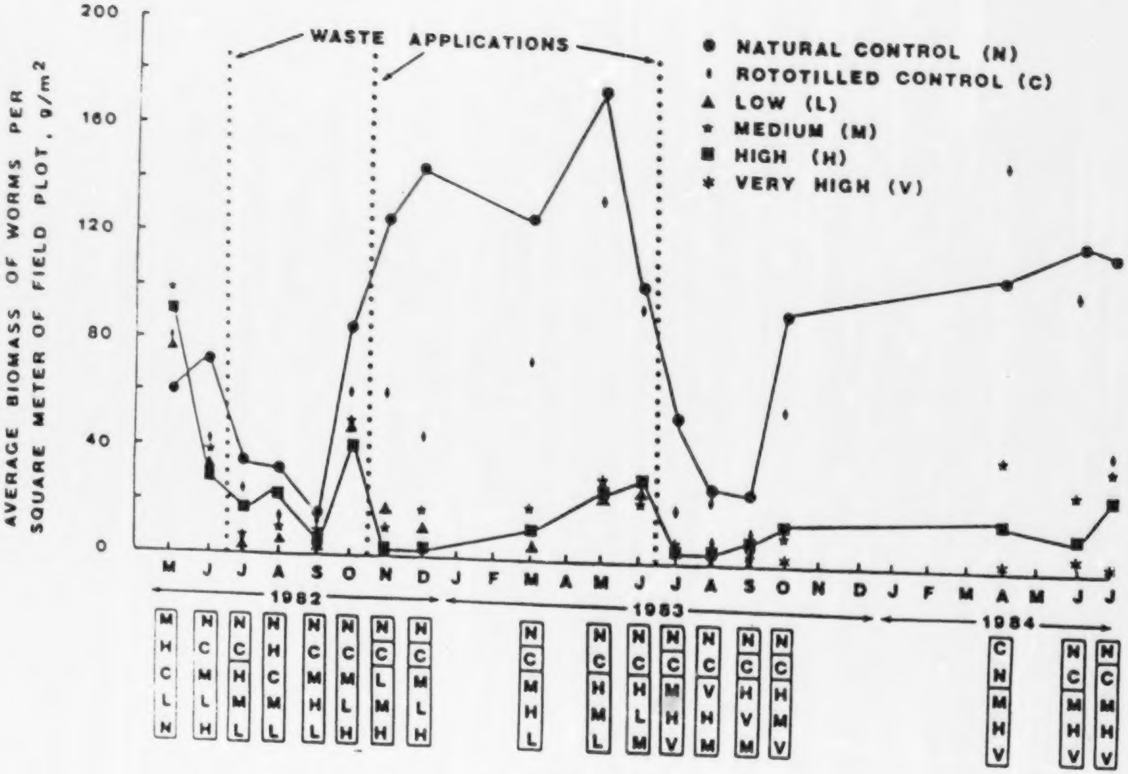


FIGURE 21
MEAN VALUES OF WORM BIOMASS FOUND IN THE FIELD PLOTS --
SYMBOLS ENCLOSED IN THE SAME BOX INDICATE THAT THE MEANS FROM THOSE
PLOTS ARE NOT SIGNIFICANTLY DIFFERENT (P = 0.05)

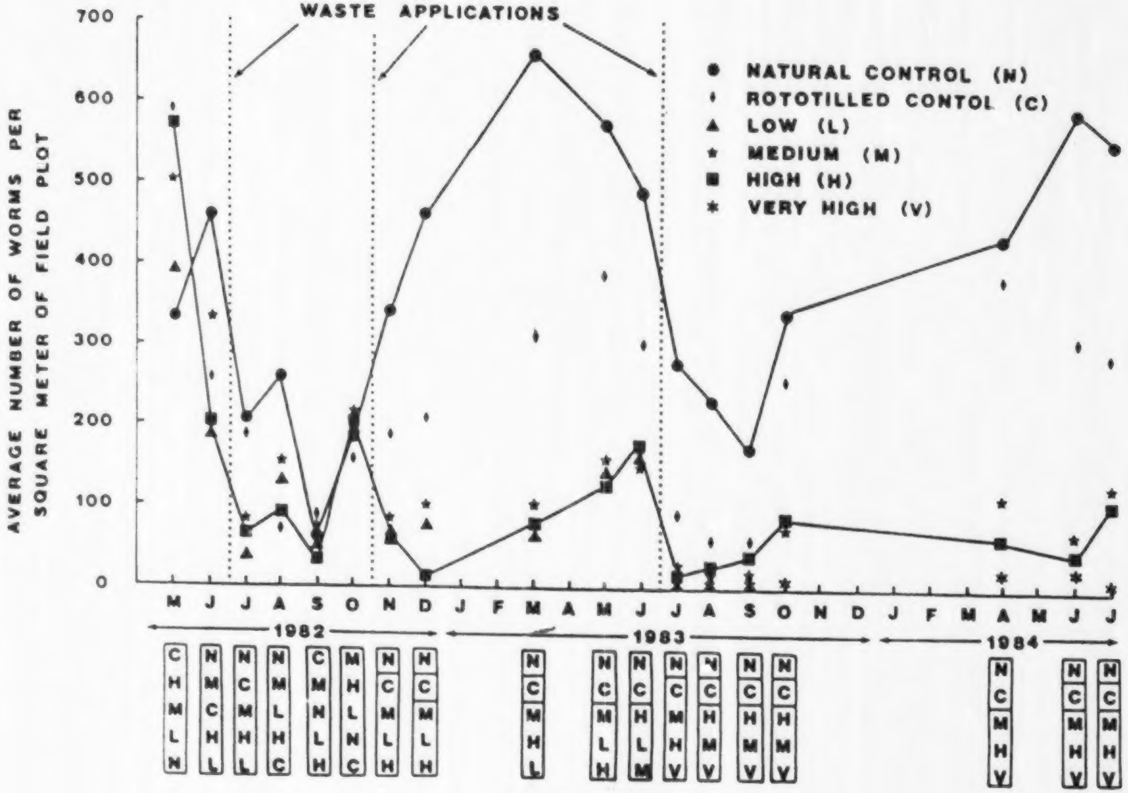


FIGURE 22
MEAN VALUES OF THE NUMBERS OF EARTHWORMS FOUND IN THE FIELD PLOTS -- SYMBOLS ENCLOSED IN THE SAME BOX INDICATE THAT THE MEANS FROM THOSE PLOTS ARE NOT SIGNIFICANTLY DIFFERENT (P = 0.05)

between the data from the control plots and from the plots that had the waste applied. When statistical differences at the 5% level ($P \leq 0.05$) were observed, Duncan's new multiple range test was used to determine where the differences were.

The differences that resulted from the statistical analysis are shown in Figures 21 and 22 in the boxes at the bottom of the figures. Where all of the types of plots are noted within one box, no statistical difference ($P \leq 0.05$) was found between the data from the plots. For example, in May 1982 (Figure 21), there were no significant differences in average biomass found in any of the type of plots. This was to be expected since at that time, no waste had been applied to any of the plots and and no rototilling had occurred.

However, after the waste was applied and rototilling occurred, there was an impact. As an example, in November 1982, after the second waste application and the second rototilling, significant differences in average biomass in the field plots were found (Figure 21). The portrayal of significance in Figure 21 for November 1982 indicates that the biomass in the natural controls was different from that in the rototilled controls and that the biomass in both types of controls were different from the biomass in the plots that had received the wastes. Furthermore, there were no significant differences in the worm biomass in the plots that had received the low, medium and high waste applications.

The statistical analyses indicated that the first waste application and rototilling had little effect on worm biomass and numbers in the field plots. However, the second rototilling and waste application in October 1982 had a significant effect on both biomass and earthworm numbers. These differences continued through the remainder of the study.

The very high waste application to the low plots in June 1983 did not seem to have any significantly different effect on the earthworms than did the previous application in October 1982. There was a trend of recovery in biomass and numbers for the medium and high application plots in 1984. However, there was not a statistically significant difference between the plots that received the wastes. This lack of significance was due in part to the large variation in biomass and numbers that occurred between plots that had the same type of waste application (medium, high or very high).

The trend toward recovery correlates with the data for the artificial test using soil from the field plots (Table 6). The loss of constituents in the waste with time reduces the apparent toxicity to the earthworm and allows the earthworms to repopulate the field plots.

The earthworm density in the natural and rototilled controls tended to be greater in the spring and fall. This is the time when moisture and temperature conditions in soil are the most favorable for earthworms in the top 15 cm of soil. This corresponds to earthworm density fluctuations shown in other studies (13, 14) in the eastern United States.

Physical disturbance of the soil, such as rototilling, can have an adverse impact on earthworms. The lower earthworm numbers and biomass in the field plots were a result of climatic conditions, rototilling and/or the applied waste.

Although rototilling did have an impact on the earthworms (Figures 21 and 22), the major impact resulted from climatic factors (temperature and soil moisture) and from the immediate effect of the waste. There was no difference in the biomass and numbers between the natural and rototilled controls until after the second application of waste and the rototilling in the fall of 1982.

Other information suggests that a single plowing of a grassland does not necessarily decrease earthworm numbers (15) although repeated cultivation of grassland can result in a decrease of earthworm populations. The second rototilling (fall 1982) did cause a significant decrease of the earthworm numbers and biomass in the rototilled control plots as compared to the numbers and biomass in the natural control plots. The data indicate that mature worms can rebound from a physical disturbance such as rototilling.

As noted earlier (Figures 14 and 15), after the waste application on June 1983, the temperatures in the soil of the very high plots were higher than that of the soil in the other plots. These higher soil temperatures (24-26°C) also probably were a contributing factor to the low earthworm populations in the very high plots. The earthworm species found in the field plots tend to avoid temperatures over 25°C.

Climatic factors such as the temperature and moisture content of soil have a large impact on the earthworm biomass and numbers found in surface soils (16). In this study, the worm biomass appeared inversely related to soil temperature (Figure 23) and appeared reasonably well correlated with soil moisture (Figure 24). The data indicated that as the temperature in the upper 15 cm of soil increased, the worm biomass in the natural controls decreased. The same relationship was found for worm numbers.

The application of the wastes had definite impacts on earthworm numbers and biomass in the field plots. The impacts were due to rototilling and the immediate effect of the applied waste. The significance of the impacts varied. However, the impacts and the pattern of change can be summarized by the conceptual model shown in Figure 25.

The earthworm population was not significantly different among plots prior to the initial rototilling and first waste application. The earthworm biomass and numbers decreased somewhat due to the rototilling and decreased more so due to the immediate effect of the applied waste. The recovery from the rototilling was fairly rapid. However, the recovery from the immediate effect of the applied waste (treatment effect) took a much longer period of time. These impacts are in addition to the natural changes in worm biomass and numbers that occur due to changes in climatic conditions (Figures 23 and 24).

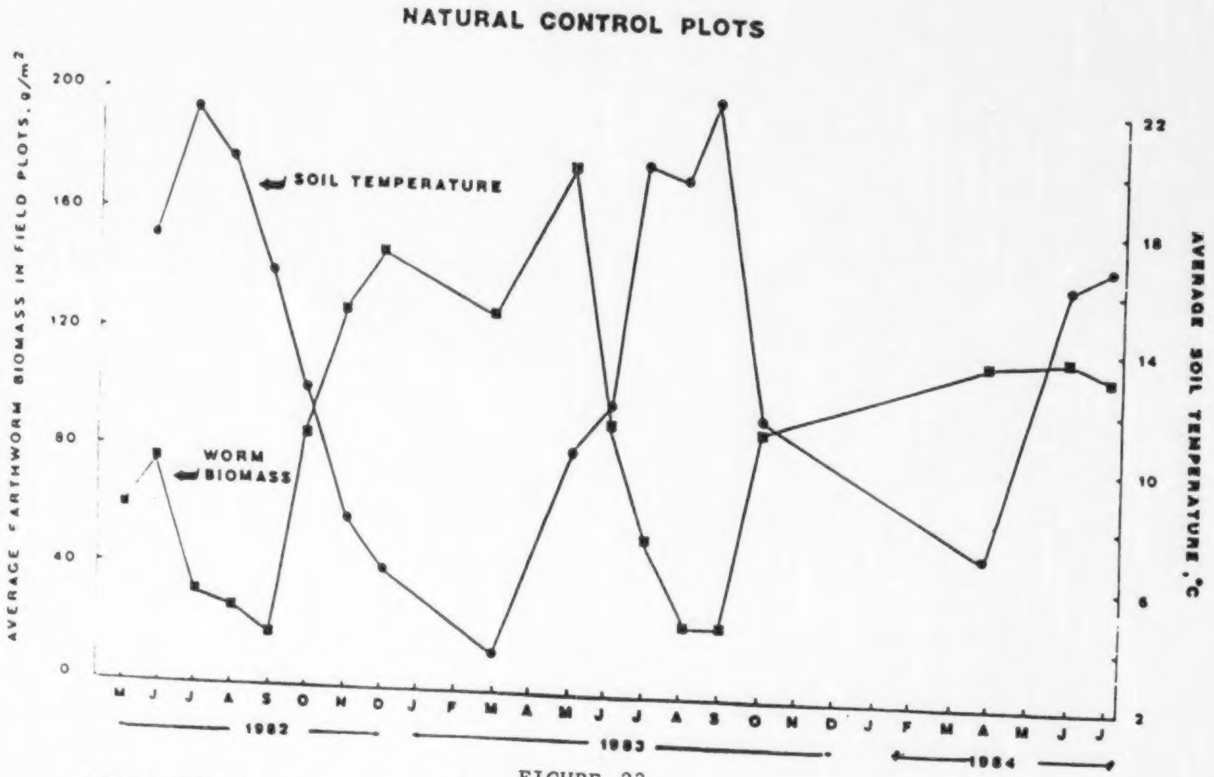


FIGURE 23
COMPARISON OF EARTHWORM BIOMASS AND SOIL TEMPERATURE IN THE FIELD PLOTS

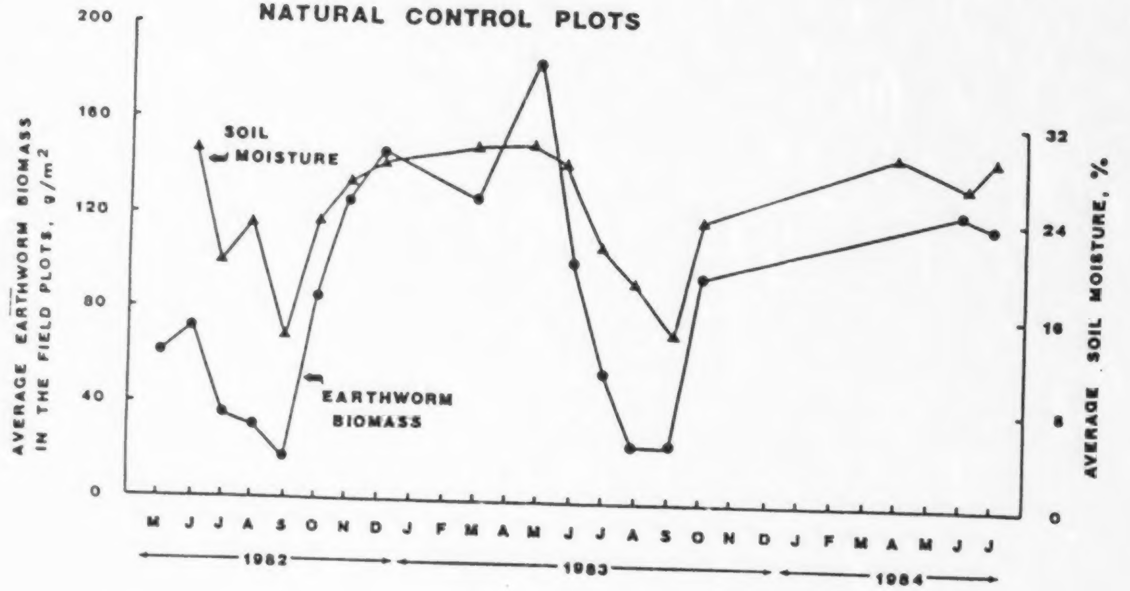
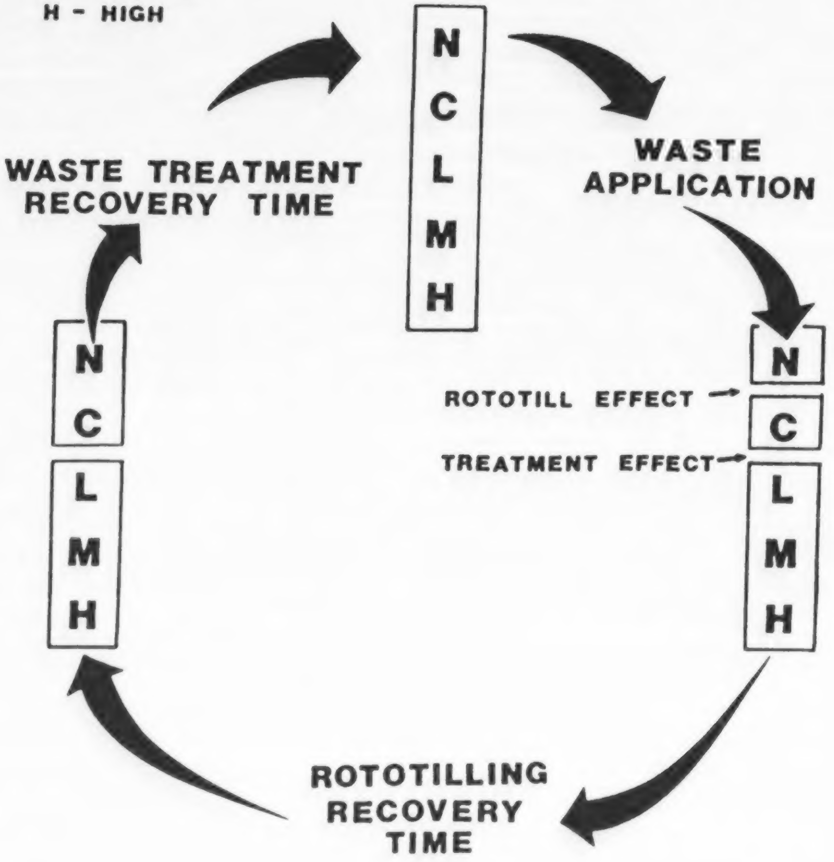


FIGURE 24
COMPARISON OF EARTHWORM BIOMASS AND SOIL MOISTURE IN THE FIELD PLOTS



N - NATURAL CONTROL
C - ROTOTILLED CONTROL
L - LOW
M - MEDIUM
H - HIGH
N C L M H
WASTE APPLICATION
N C L M H
ROTOTILL EFFECT
TREATMENT EFFECT
ROTOTILLING RECOVERY TIME
N C L M H
WASTE TREATMENT RECOVERY TIME

FIGURE 25
A GENERAL CONCEPTUAL MODEL OF THE IMPACT TO AND RECOVERY OF SOIL BIOTA WHEN WASTES ARE APPLIED TO THE SOIL

The results suggest that soil biota such as earthworms can recover from single, even large applications of an oily waste of the type used in this study. With time and no other impacts, the earthworm population in all the field plots should again become similar to that in the natural control plots.

Bioaccumulation

The bioaccumulation of metals and organics in an applied waste by earthworms is of environmental interest because the worms can serve as a food source for higher forms of life such as mice, moles and other small mammals and birds. The concentrations of metals and organics in the soil and the worms were analyzed to determine if any bioaccumulation resulted from the application of the wastes. The concentrations of metals found in the worms are summarized in Appendix J.

In analyzing the metal and organic content of the worms, the entire worm was used including the content of the gut. This was done since it is the entire worm that may be eaten by birds and small animals. Because the interest was to identify the bioaccumulation that might occur and the potential impact on other parts of the ecosystem, it was appropriate to consider the entire worm. The worms were washed, however, to remove any soil or other material that may have adhered to the skin.

The gut content of the worm will include some of the soil surrounding the worm. In interpreting the bioaccumulation data, it should be recognized that the data represent both earthworm biomass and the soil and other material in the gut of the earthworm.

Metals - The concentrations of metals found in the earthworms on the noted sampling dates were not as extensive as that of the soil metal concentrations because, on many of the sampling dates, there were few or no worms.

The data for several metals (chromium, copper, lead and zinc) that are of potential environmental concern were evaluated to determine if there were any statistically different concentrations in the worms as a result of the waste applications. Example results are presented in Table 17 for chromium and zinc and in Table 18 for copper and lead.

In June 1983, the low application plots became the very high application plots (SECTION 5). However, because of the very high waste application of June 1983, worms were not found in the very high plots for a considerable period of time after the application. Data on the metal concentrations in the earthworms of the very high plots were obtained only in June 1984. In the statistical analyses (Tables 17 and 18), the very high worm metal concentrations were included with the low application data to see if any differences could be ascertained.

The analyses indicate that only the chromium concentration of the earthworms increased as a result of the waste applications. Except for chromium, the concentrations of copper, lead and zinc in the worms of the

TABLE 17. AVERAGE CHROMIUM AND ZINC CONCENTRATIONS[+] IN THE EARTHWORMS OF THE FIELD PLOTS — STATISTICAL ANALYSIS[*]

Plots	Date of Sample[++]						
	6/2/82	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Chromium							
Natural Control	9.7^a	7.8^a	10.2^a	8.3^a	11.0^a	12.5^a	10.0^a
Rototilled Control	7.4^a	15.3^a	13.3^a	8.5^a	16.0^a	12.3^a	11.5^a
Low Application	6.2^a	13.8^a	11.4^a	8.0^a	30.0^a	ND	37.9^{a}**
Medium Application	6.8^a	10.7^a	12.7^a	10.8^a	18.0^b	20.0^b	18.8^b
High Application	8.2^a	7.4^a	19.2^b	ND	21.0^b	18.5^b	21.5^b
Zinc							
Natural Control	222^a	258^a	223^a	238^a	290^a	269^a	258^a
Rototilled Control	305^a	278^a	183^a	350^a	250^a	221^a	232^a
Low Application	255^a	323^a	248^a	225^a	300^a	ND	866^{a}**
Medium Application	225^a	241^a	243^a	433^a	230^a	218^a	279^a
High Application	225^a	248^a	220^a	240^a	198^a	202^a	387^a

+ mg/kg moisture free earthworm tissue and gut contents; the number of worms collected and analyzed on each sampling date were not constant - on many sampling dates, few worms were found; ND = below detection limits for the quantity of sample obtained on that date or else no worms found.

* data with the same superscript in a horizontal row are not statistically different at the 95% confidence level ($P<0.05$).

++ waste applications were in mid-June 1982, late October 1982 and mid-June 1983.

** In June 1983, the low application plots became the very high application plots (see text, SECTION 5).

TABLE 18. AVERAGE COPPER AND LEAD CONCENTRATIONS$^+$ IN THE EARTHWORMS OF THE FIELD PLOTS — STATISTICAL ANALYSIS*

Plots	Date of Sample^{++}						
	6/2/82	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Copper							
Natural Control	11.8^a	11.4^a	11.8^a	12.2^a	12.0^a	10.3^a	10.7^a
Rototilled Control	12.0^a	28.6^a	13.0^a	11.0^a	12.0^a	11.0^a	10.5^a
Low Application	11.2^a	20.0^a	13.0^a	16.7^a	26.0^a	ND	14.3^a**
Medium Application	9.6^a	16.1^a	36.3^a	11.5^a	17.0^a	12.0^a	12.0^a
High Application	11.5^a	12.2^a	15.5^a	ND	12.0^a	14.5^a	13.5^a
Lead							
Natural Control	12.0^a	8.5^a	12.7^a	14.5^a	8.6^a	8.8^a	7.8^a
Rototilled Control	10.5^a	6.3^a	14.7^a	15.1^a	13.2^a	8.8^a	8.0^a
Low Application	9.8^a	9.0^a	16.3^a	15.0^a	13.5^a	ND	ND **
Medium Application	8.0^a	19.0^a	16.3^a	12.0^a	14.3^a	ND	10.3^a
High Application	8.3^a	10.5^a	18.0^a	ND	15.5^a	15.0^a	10.3^a

$^+$ mg/kg moisture free earthworm tissue and gut contents; the number of worms collected and analyzed on each sampling date were not constant - on many sampling dates, few worms were found; ND = below detection limits for the quantity of sample obtained on that date or else no worms found.

* data with the same superscript in a horizontal row are not statistically different at the 95% confidence level ($P<0.05$).

$^{++}$ waste applications were in mid-June 1982, late October 1982 and mid-June 1983.

** In June 1983, the low application plots became the very high application plots (see text, SECTION 5).

control plots were statistically the same as that of worms of the plots that received the waste applications.

In a similar manner, the concentration of all of the metals were compared using a one way ANOVA to identify if there were any significant differences in the earthworm concentrations of other metals over the entire project period. The comparison is presented in Table 19. The analysis indicates that there were several situations in which there were significant differences. There was no obvious pattern to the differences however. Earthworms from the rototilled controls had significant differences of aluminum, iron, nickel and vanadium whereas the worms from the high application plots had significant differences for aluminum, chromium, and titanium. Because of the lack of a pattern, such as differences always showing up in the plots with the higher waste applications, it may be that the differences noted in Table 19 are due to normal differences in metal concentrations in earthworms.

The bioaccumulation of metals that occurs is of interest since worms are a part of the food chain. Table 20 summarizes the range of metal bioaccumulation that was found in the earthworm tissue. In this table, bioaccumulation is defined as the ratio of the metal concentration in the earthworm to the metal concentration in the soil of the plot from which the earthworm was obtained. Bioaccumulation factors greater than one indicated that the metal was being selectively accumulated by the earthworm. Factors slightly over one should not be considered important because of the data variations that may have been caused by sampling and analytical variations and because this factor is a ratio. Bioaccumulation factors that are much larger than one are relevant and indicate that bioaccumulation has occurred.

Based on the data in Table 20, the earthworms accumulated cadmium, potassium, sodium and zinc. Potassium and sodium are of physiological but not environmental importance in terms of bioaccumulation.

Cadmium bioaccumulates in earthworms to a greater degree than any other metal. The bioaccumulation factor of cadmium in earthworm tissue rarely was less than two and commonly was greater than 20.

For comparative purposes, the bioaccumulation of cadmium, copper, zinc and lead by earthworms as found by other investigations is summarized in Table 21. Because of the large amount of information available in each paper, the ranges of bioaccumulation data are indicated. As noted from Table 21, the bioaccumulation data obtained in this study were similar to that reported by other investigators.

The bioaccumulation factors for other metals can be compared to factors identified in a recent comprehensive study (Table 22). Generally the factors in Table 22 are less than those reported in this study.

TABLE 19. STATISTICAL EVALUATION+ OF THE EARTHWORM METAL CONCENTRATIONS DURING THE PROJECT PERIOD (F VALUES)

Metal	Natural Control Plots	Rototilled Control Plots	Low++ Application Plots	Medium Application Plots	High Application Plots
Al	1.87	12.3*	2.47	2.38	5.82*
Ca	0.50	2.16	2.20	2.10	2.70
Cd	1.24	1.40	2.16	0.85	0.83
Cr	1.86	0.55	2.92	4.80*	3.74*
Cu	0.53	0.84	1.35	1.19	1.24
Fe	1.14	10.9*	3.46*	1.52	2.71
K	1.10	1.60	1.02	1.16	1.85
Mg	0.68	2.17	4.45*	2.48	2.24
Mn	0.81	1.94	1.78	1.01	0.88
Na	1.43	1.80	3.99*	0.87	2.00
Ni	2.39	5.92*	0.89	3.49*	2.39
Pb	1.87	2.05	1.62	1.17	2.69
Ti	1.98	1.87	7.93*	2.35	6.25*
V	3.13*	8.46*	3.12	5.47*	3.03
Zn	0.59	1.15	2.54	1.06	2.82

+ One way ANOVA using all of the earthworm data from the respective plots (Appendix).

++ In June 1983, the low application plots became the very high application plots (see text, SECTION 5). The data for 6/6/84 from the very high plots was included with the data from the low plots.

* indicates significance at the 5% level ($P \leq 0.05$)

TABLE 20. BIOACCUMULATION[+] OF METALS BY EARTHWORMS[++]

	Date of Sampling				
Metal	June 1982	November 1982	June 1983	October 1983	June 1984
Al	0.12-0.29	0.24-0.30	0.30-0.43	0.34-0.42	0.11-0.37
Ca	0.70-1.20	0.62-1.70	0.48-1.12	0.98-1.12	0.45-1.04
Cd	8.0-14.4	5.9-11.5	14.0-29.6	NA*	6.6-10.3
Cr	0.20-0.30	0.16-0.34	0.26-0.58	0.45-0.51	0.25-0.42
Cu	0.75-1.91	0.55-0.79	0.48-1.73	0.65-0.79	0.42-0.72
Fe	0.21-0.36	0.30-0.51	0.33-0.46	0.37-0.44	0.20-0.40
K	1.70-2.65	2.03-3.73	0.97-2.13	1.01-2.42	0.89-3.06
Mg	0.36-0.45	0.43-0.59	0.46-0.56	0.49-0.58	0.33-0.49
Mn	0.18-0.34	0.26-0.46	0.28-0.43	0.34-0.42	0.23-0.41
Na	10.3-16.6	4.5-8.7	9.6-21.6	23.0-32.0	6.7-67.7
Ni	0.23-0.54	0.41-0.48	0.64-0.77	0.46-0.70	0.35-0.73
Pb	0.30-0.70	0.38-0.60	0.36-1.04	0.45-0.68	0.37-0.43
Ti	0.23-0.83	0.28-0.42	0.50-1.01	0.60-0.85	0.45-0.86
V	0.19-0.38	0.20-0.29	0.36-0.47	0.44-0.57	0.30-0.39
Zn	1.20-2.10	1.5-3.7	0.99-2.95	1.81-2.92	1.66-2.55

+ Ratio of the metal concentration in the earthworm to the metal concentration in the soil of the plots from which the earthworms were obtained.

++ Range of data from soil and worm samples collected on noted dates; numbers represent the range of mean values (n = 4) from each type of plot (controls, low, medium, high, very high).

* NA - not available, not enough tissue available for analysis.

TABLE 21. EARTHWORM BIOACCUMULATION FACTORS FOR CADMIUM, COPPER, LEAD AND ZINC.*

Metal				Reference
Cadmium	Copper	Lead	Zinc	
--	--	0.2	--	17
11.6-22.5	--	--	3.1-13.4	18
7.0-15.2	--	0.5-1.2	2.2-8.1	19
11.6-22.5	--	0.1-0.3	3.1-13.5	20
--	--	0.8-2.4	0.3-0.7	21
2.9-12.6	--	--	--	22
--	--	0.9-1.3	1.6-2.4	23
3.8-5.0	--	0.4	0.5	24
--	0.4-1.5	--	--	25
17.1-31.1	0.6-0.8	0.4-0.5	7.3-17.5	26
8.8-151.4	--	0.1-0.8	--	27
1-7.5	--	0-2.7	0.7-5.4	28
4.6-6.3	0.2-0.3	--	2.2-3.7	29
1.8-15.0	0.3-4.9	1.4-9.2	--	30
3.9-35.0	--	0.2-0.5	1.0-10.3	31
18-156	--	--	1.4-4.1	32

*Data from other investigations.

TABLE 22. EARTHWORM BIOACCUMULATION FACTORS FOR SEVERAL METALS*

Metal	Range of Reported Data	Metal	Range of Reported Data
Ca	0.4-0.7	K	0.40-0.53
Cr	0.03-0.11	Mn	0.02-0.22
Fe	0.02-0.25	Na	0.59-0.88

*From reference 32

The data indicate that, of all the metals of potential concern, cadmium bioaccumulates to a greater extent. Only the very high waste application increased the cadmium concentration of the soil significantly (Tables 11 and 12). The cadmium that accumulated in the earthworm tissue probably came more from the background cadmium in the soil than from the cadmium in the applied waste, especially since the cadmium bioaccumulation occurred at comparably high levels in the worms from the control plots.

Increased concentrations of chromium, copper, lead and zinc occurred in the soil as a result of the waste applications (Table 12). However, none of these metals bioaccumulated in earthworms to any greater extent than was found in other studies (Tables 21 and 22) or in earthworms from the control plots.

Thus, it appears that the application of these oily wastes did not cause any abnormal or unexpected bioaccumulation of metals in earthworms. It is also apparent that earthworms bioaccumulate few of the metals found in soils.

Organics - The earthworm sample extracts were analyzed for the same organic compounds that were determined for the soil extracts (Table 16). None of these compounds were found in any of the earthworm extracts at concentrations greater than the detection limits for these compounds. The detectable limits were 10 ng/ul when the analytical methods at RSKERL were used. These results suggest that earthworms do not accumulate any of the noted organic compounds.

MICROARTHROPODS

Soil animals are an integral part of any soil system and play a major role in the initial formation of soils and in the continued decomposition of organics. Investigations of soil microarthropods, primarily of mites and collembolans, have become increasingly important in the study of ecosystem perturbations. These microarthropods are: (a) numerous, with usually several hundreds of thousands of individuals per square meter in non-cultivated soils, (b) taxonomically diverse, with 100 to 200 species represented in habitats such as old fields, (c) trophically diverse and include species which are predaceous, parasitic, fungivorous or detritivorous, (d) relatively easy to collect and preserve and (e) significant functional components of soil communities.

Four principal groups of soil microarthropods were examined. One was the insect order Collembola (collembolans or springtails) which feed principally on soil fungi or organic detritus, although a few are facultative or obligatory predators of soil nematodes or other soil organisms. The other three were suborders of mites which are important in soils. These are the Oribatida (detrivores or fungivores); the Mesostigmata (predators of Collembola, other mites, or soil nematodes); and the Prostigmata (heterogeneous, fungivores, predators or parasites of other soil animals). These soil organisms were chosen because they are numerically dominant in soil ecosystems.

Soil samples containing microarthropods were collected only during the first project year. The sampling dates are noted in Table 23. Three cores were removed from each plot on each sampling date for a total of sixty samples on each date. The sampling points were determined at random and were different from those used for the earthworm and soil samples. No subplots were sampled more than once. To avoid trampling effects, all subplots were sampled from a portable wooden spanning bridge. Cores were not directly handled. They were left in the aluminum corer and placed into individual plastic bags which were then deposited in styrofoam ice-chests. Extraction began within three hours of collection.

The procedures used to extract and identify the microarthropods are presented in Appendix A.

For analyses using parametric statistics such as ANOVA and multiple comparison tests, the numbers of individuals per sample for each species were subjected to a log-transformation ($n_t = \log_{10}(n+1)$) prior to analysis. This is a standard technique (33, 34) for eliminating the problem of having sample variances which change with each sample (heterogeneity of

TABLE 23. SOIL MICROARTHROPOD SAMPLING DATES

1982	
June 2	September 13
June 9-10	October 12
June 22	October 22-23
July 14	November 12
August 13	December 2
1983	
March 15	
June 8	
July 8	

variance), a condition which violates an important assumption of parametric tests. This technique "normalizes" the data without changing its information content. Heterogeneity of variance is inherent in analyzing the density of most organisms because of their aggregated, rather than random spatial distributions. This is especially true of microarthropods and other soil organisms (35, 36). Transformed means can be returned to numbers reflecting "real individuals" by taking the antilog of the mean and subtracting one.

A one-way analysis of variance (ANOVA) procedure was performed at each sampling period to test the hypothesis that there was no difference between means for the various treatments and controls. When statistical significance differences ($P \leq 0.05$) were noted, the ANOVA was followed by a Student-Newman-Keuls (SNK) multiple comparison test to determine which pairs of treatments were significantly different with regard to mean density of that particular species. Similar analyses were performed for certain higher taxonomic groups, in which the data for individual species were pooled.

Results - The pooled numbers for the four major groups during all sampling periods are given in Table 24. Overall, Prostigmata were the most prevalent microarthropods sampled, while the Mesostigmata were the lowest in density. In terms of average number of microarthropods per meter^2, there were fewer organisms in the plots that were tilled and had the wastes applied. In general, as the application rate increased, the number of organisms per meter^2 decreased. Table 25 expresses these overall decreases as percentages of natural control plot results.

Figure 26 illustrates the total microarthropod pattern as a function of time. The impact of rototilling and the oil waste appears temporary. The total microarthropod density in the natural control plots varied little over time. There were no significant differences between plots before the June 1982 application. However, there was a significant rototilling and treatment effect after the June and October applications.

The rototilling effect from the June 1982 application was short with recovery occurring after one month. The rototilling effect from the October 1982 application was equally strong but the organisms did not recover until some time between March and June 1983. This slow recovery probably was due to the cold weather after the October 1982 treatment.

Following the June 1982 rototilling and waste application, recovery of microarthropod densities was gradual but complete within four months. By mid-October 1982, all plots had total microarthropod densities similar to those of natural controls (Figure 26). The October 1982 rototilling and waste application effects were initially equally as great, but recovery was slower because of the colder temperatures during the winter of 1982-83 and was not complete when the microarthropod evaluation ended in July 1983.

The impact of rototilling and waste application on the dominant species of microarthropods is summarized in Table 26. Although

TABLE 24. TOTAL NUMBERS OF MICROARTHROPODS COLLECTED AT THE FIELD PLOTS DURING JUNE 1982 TO JULY 1983

	Collembola	Mesostigmata	Oribatida	Prostigmata	Total	Average Number per meter^2
Natural Control	2,740	688	2,770	6,340	12,538	58,000
Rototilled Control	1,490	485	2,340	5,510	9,825	46,900
Low Application	572	327	538	1,480	2,917	13,200
Medium Application	258	294	257	1,730	2,539	11,600
High Application	150	438	262	1,090	1,940	8,840
Total	5,210	2,232	6,167	16,150	29,759	138,540

TABLE 25. MICROARTHROPODS IN THE FIELD PLOTS EXPRESSED AS % OF NATURAL CONTROL PLOT NUMBERS*

	Collembola	Mesostigmata	Oribatida	Prostigmata	Total
Natural Control	--	--	--	--	--
Rototilled Control	54%	70%	84%	87%	78%
Low Application	21%	48%	19%	23%	23%
Medium Application	9%	43%	9%	27%	20%
High Application	5%	64%	9%	17%	15%

*Data from Table 24

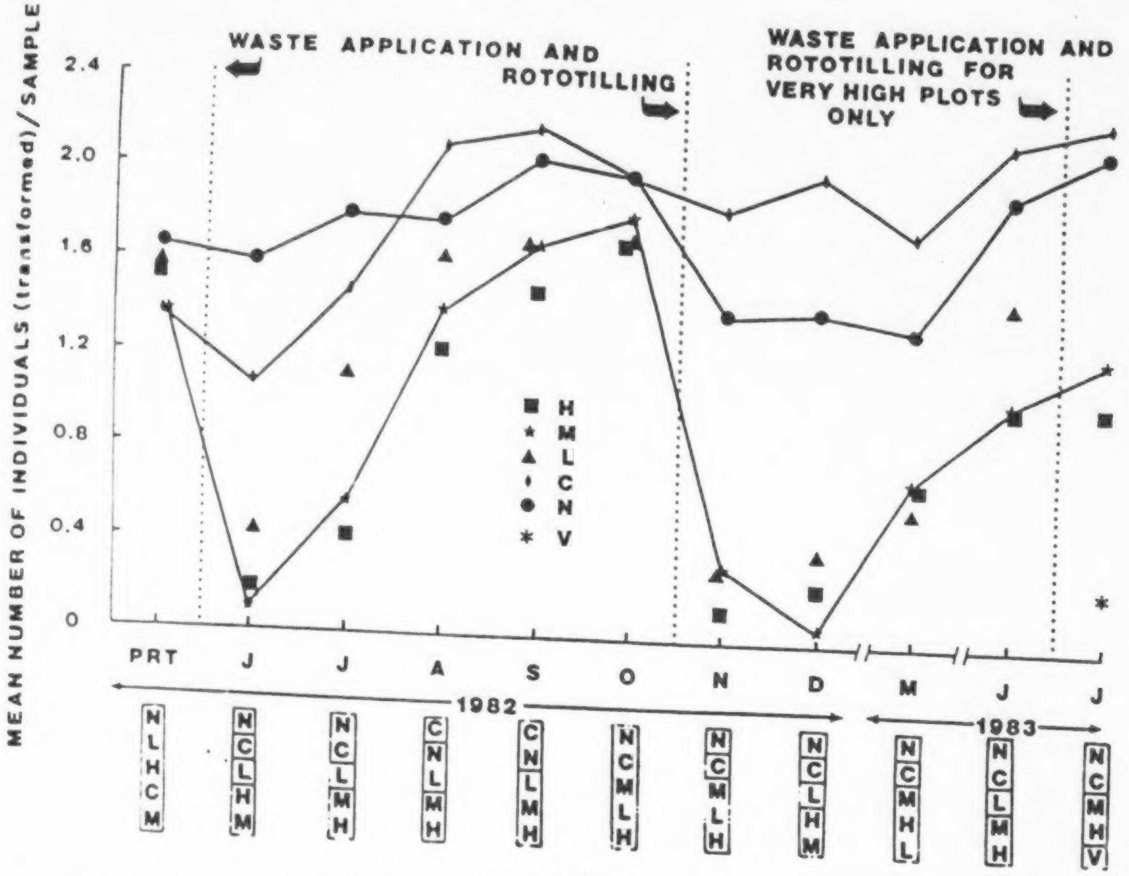


FIGURE 26
MEAN TOTAL MICROARTHROPODS (MITES AND COLLEMBOLANS) COLLECTED AT THE FIELD PLOTS -- DATA TRANSFORMATION IS LOG_{10} (n + 1) -- SYMBOLS ENCLOSED IN THE SAME BOX INDICATE THAT THE MEANS FROM THOSE PLOTS ARE NOT SIGNIFICANTLY DIFFERENT (P = 0.05)

TABLE 26. IMPACT OF ROTOTILLING AND OILY WASTE APPLICATION ON MICROARTHROPOD SPECIES IN FIELD PLOTS

	First Application (June 1982)		Second Application (October 1982)	
	Rototill Effect	Waste Effect	Rototill Effect	Waste Effect
COLLEMBOLA				
Isotoma notabilis	-(d)	-(m)	-(d)	-(i)
Isotomiella minor	+(d)	0	-(i)	-(m)
Folsomia fimetaria	-(i)	0	-(i)	0
Isotomurus palustris	+(d)	0	-(d)	0
ONYCHIURIDAE (pooled)	-(d)	0	-(i)	0
ENTOMOBRYIDAE (pooled)	-(i)	0	-(d)	-(i)
SMINTHURIDAE (pooled)	-(i)	-(i)	-(i)	-(i)
ORIBATIDA				
Scheloribates laevigatus	-(i)	-(m)	0	-(i)
Tectocepheus velatus	-(i)	-(m)	-(i)	-(m)
Punctoribates armipes	+(d)	0	0*	0
Oppiella nova	0	0	+(d)	-(d)
BRACHYCHTHONIIDAE (pooled)	-(d)	0	-(d)	0
PROSTIGMATA				
Scutacarus sp. A	-(i)	0	-(i)	0
Bakerdania sp. A	-(i)	0	-(i)	0
Bakerdania sp. E	+(d)	0	+(i)	0
Tarsonemus sp.	-(i)	-(m)	-(d)	-(i)
Benoinyssus sp. A	-(i)	-(m)	-(i)	-(i)
Cocceupodes sp. B	0	-(m)	-(i)	-(m)
Tydeus sp. A	-(d)	0	-(d)	-(i)
Microtydeus sp.	0	-(d)	-(d)	-(i)
Coccotydeus sp.	+(d)	-(d)	0	-(d)
Alicorhagia sp.	0	0	-(d)	-(d)
MESOSTIGMATA				
Paragamasus sp.	-(d)	0	-(i)	-(m)
Rhodacarus sp.	0	0	0	0
Rhodacarellus sp.	-(d)	0	-(d)	0
Arctoseius cetratus	+(d)	+(d)	+(i)	-(i)

+ = density significantly (P = .05) increased.
− = density significantly (P = .05) decreased.
0 = no significant change.
i = immediate effect.
d = delayed appearance of effect due to prior (seasonal) absence of species.
m = delayed appearance of treatment effect due to strong rototill effect.
* = densities were higher in Control plots, but not significantly so.

rototilling decreased the numbers of most species, it had a positive effect on a few species. The application of the waste had an almost universal negative impact on the microarthropods.

The land treatment of the wastes and the climatic conditions had an effect on the soil microarthropod community that was similar to that noted for the earthworms. Soil temperature and moisture, rototilling, and the applied waste all had an effect on the microarthropod population. The significance of these impacts, and the time necessary for recovery varied with both the microarthropod population being measured and with the taxonomic group. The conceptual model presented for the earthworms (Figure 25) also can be applied to the microarthropod data. The microarthropod density was not significantly different between plots prior to rototilling and waste application. The population was decreased by the rototilling and decreased even more so by the waste application (treatment effect). The recovery from the rototilling generally preceded recovery from the treatment effect. With time and no other impacts, the microarthropod population should become similar for all the field plots.

Negative impacts of cultivation on soil microarthropods have been noted by a number of workers, mostly in Europe (37-39). Two reasons for the negative impacts have been proposed. First, physical abrasion suffered during cultivation may damage or kill the animals. This is much more likely to be a problem with larger animals, such as earthworms. A second, and intuitively more important probable cause of observed decreases, is the production of unstable microclimates. Removal or incorporation of the thick vegetation and litter, characteristic of pastures and old-fields, eliminates an effective natural insulator. Marked temperature, pH and moisture fluctuations did occur in this study (Figures 10, 14 and 16). Microarthropods are generally adapted to living in a highly structured, spatially heterogeneous physical environment with a relatively homogeneous microclimate. As a result of cultivation (rototilling), this relationship is reversed making the soil more homogeneous and the microclimate heterogeneous and unstable.

The reason for the immediate effect of the wastes on microarthropods is not clear. Clear negative impacts on community structure were noted for all major groups except the Mesostigmata, and the possible reasons for these impacts are several. First, there may be a direct toxicity to the animals. If any microarthropods were to show toxicity it would probably be the Mesostigmata, which are active predators utilizing larger volumes of soil in their movements than other mites. However, they are the one group which was not significantly affected by the waste application.

The immediate impact of the waste appeared to be independent of the application rate. Densities and other community parameters were generally depressed as much by low treatments in June 1982 as by the very high treatment in June 1983, which was about 56 times greater. The tests for significance also revealed few taxa or times when the various rates had different effects.

In summary, the application of the oily wastes to an old-field site in central New York State had a predominantly negative impact on the soil microarthropods. This impact was due to both the application method (rototilling) and to the immediate effect of the oily waste.

The negative impact of rototilling was temporary, with recovery of the microarthropod population occurring within 1-3 months after late spring rototilling and between 6-8 months after the fall rototilling. The negative impacts of the oily waste were more long-lived. Total density recovery did not occur during the time frame of the study. Multiple applications per year would probably keep the system in a highly disrupted state.

Microarthropod densities were affected by the waste application. However, differences between application rates were observed mainly in the recovery with lower waste applications allowing faster recovery.

Perhaps the most significant general result of this aspect of the project is that an important group of soil organisms (microarthropods) does recover from modest inputs of oily wastes. When use of a land treatment site is discontinued, "reclamation" by soil microarthropods is likely to occur, although at a rate not presently predictable.

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APPENDIX A

MICROARTHROPOD EXTRACTION AND IDENTIFICATION PROCEDURES

The aluminum corer containing the soil sample was inverted and heat from a 7 watt light bulb was applied from above. As drying proceeds vertically down the core, a critical relative humidity is reached at which point the microarthropods travel to the bottom end of the core (originally the soil surface) where they fall into a vial filled with preservative (70% ethanol). The extraction occurred in a temperature-controlled room with an ambient temperature of 5°C. Extractors were left undisturbed for one week. Even the wettest samples had dried by this time. Most of the microarthropods emerged during the first 2 or 3 days of extraction. Vials containing preserved microarthropods were stored until the remaining steps could be accomplished.

The microarthropod identification steps were highly labor-intensive. The contents of each vial were carefully sorted under magnifications of 10 to 40 diameters to separate microarthropods from small particles of soil and organic debris which invariably fall into the preservative. Specimens were sorted into four major taxonomic categories: collembolans, mesostigmatid mites, prostigmatid mites, and oribatid mites. The first three groups were mounted on microscope slides for counting and identification. Because of low diversity and high familiarity, the oribatid mites were processed in alcohol. Identifications were made at the species level. If a species could not be identified, it was given an arbitrary letter designation.

APPENDIX B

METHODS* USED TO PREPARE SOIL AND EARTHWORM SAMPLES FOR ANALYSIS BY RSKERL

A. Extraction Procedure For Soil Organics

- Ten grams of the "as is" soil were mixed with ten grams of anhydrous Na_2SO_4
- The mixture was extracted in acid washed glass equipment for two hours or at least 20 cycles with 200 ml methylene chloride, concentrated to about 2 to 3 ml and shipped to RSKERL in glass vials.

B. Preparation For Soil Metal Analyses

- Oven dried soil samples were ground to 20 mesh.
- About 100 grams of each sample were shipped to RSKERL in glass bottles.

C. Preparation For Earthworm Metal Analysis

- Earthworm tissue samples were oven dried at 60°C for 24 hours, finely ground using porcelain equipment and shipped to RSKERL in glass bottles.

D. Extraction Procedure for Organics in Earthworm Tissue

- Fresh earthworm tissue was mixed with ten grams of anhydrous Na_2SO_4 and extracted with 200 ml of methylene chloride for two hours. The extract was concentrated to about 2 to 3 ml and shipped to RSKERL in glass vials.

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*Details of the methods were supplied to project investigators by the RSKERL project officer.

APPENDIX C

SPATIAL VARIABILITY OF THE SOIL SAMPLES FROM THE FIELD PLOTS

Introduction

This study was undertaken to determine the extent to which the variability of the data was due to the non-homogeneity of waste application and rototilling at the field plots. At each plot, three random core samples were taken from the plots periodically for analysis (SECTION 3). In addition, every attempt was made to use plots with comparable soil characteristics, to distribute the wastes uniformly, and to mix the waste and soil completely. Initially, each plot was mowed and raked to remove the existing vegetation. The plots were then rototilled to an average depth of about 15 cm to facilitate subsequent waste incorporation. Before application, the oily waste was thoroughly mixed to assure uniformity. The waste was applied to the plots by a hand-held sprayer in as uniform a manner as possible.

A split application approach was used. The total quantity of waste applied to each plot was divided into three equal parts. Following each waste application, the plots were rototilled immediately. Thus, each plot was rototilled three times during the application of the waste.

In addition, each plot was rototilled after completion of the waste application process. At this time, the plots were rototilled twice in perpendicular directions. The plots received no further tilling for the duration of the study.

Spatial Variation

Shortly after the oily waste was applied to four of the plots and the plots were rototilled in June 1983, ten random samples were taken from the zone of incorporation (ZOI) of two of the plots that received the very high waste applications. Each sample was analyzed for oil and grease, volatile material, total Kjeldahl nitrogen, and pH. Ten random samples also were taken from the same plots in November 1983. The second set of samples helped identify the effects of time and degradative processes on the spatial variation. The November samples were analyzed for the four parameters noted above and for moisture content.

Each soil sample was kept separate and was prepared for analysis in the following manner. First, each sample was mixed and hand sorted to remove stones and large vegetative material. After subsamples were taken for moisture and pH determinations, the remaining soil was air dried at room temperature (about 20°C). When air dry, the samples were pulverized and passed through a No. 16 U.S. Standard Sieve which had a mesh opening of 1.19 mm. The screened samples were then stored in screw-cap bottles at room temperatures until analyzed.

The results of the June and November 1983 analyses are summarized in Tables C-1 and C-2. There were substantial variations in soil characteristics, particularly in oil and grease, among each set of samples even though every effort had been made to obtain the most uniform soil-waste mixture possible.

Figure C-1 illustrates the typical distribution of the parameters in the subplot samples. Such distributions were similar for both sets of subplot soil samples.

In each set, there were substantial differences among subplot samples for all of the parameters considered with the exception of pH (Tables C-1 and C-2). The observed differences could be due to either spatial variation or random analytical error. To separate these factors, the analytical results from each set of 10 subplot samples, two replicates per sample for each parameter, were compared statistically.

The comparisons were made using a one way analysis of variance (8) to test the null hypothesis that the subplot means for each parameter do not differ significantly. The results of the comparison (Table C-3) indicated that the observed differences within each set of subplots were statistically significant ($P<0.05$), and were due to spatial variation and not random error.

The observed differences in mean values for oil and grease, total Kjeldahl nitrogen, and volatile material between plots and between sampling dates for each plot also were tested for statistical significance. These comparisons were made using one-way analysis of variance (8) followed by Duncan's new multiple range test (9) to analyze differences between means.

The results of these statistical analyses are summarized in Table C-4. Oil and grease concentrations in the two plots were not significantly different ($P<0.05$) in either June or November. However, the oil and grease concentrations were significantly different between June and November indicating that reductions did occur in both plots between those months.

There were significant differences between plots in total Kjeldahl nitrogen and volatile material concentrations in both June and November but no significant changes occurred in either plot with time. Although significant reductions in oil and grease occurred in both plots between June and November, the spatial variation as indicated by the coefficient of variation for each plot (Tables C-1 and C-2) remained essentially constant.

TABLE C-1. SUMMARY OF THE SOIL ANALYSIS - JUNE 1983.

		Oil and Grease (g/kg MFS*)	Volatile Material (% MFS*)	Total Kjeldahl Nitrogen (g/kg MFS*)	pH
Plot I	Range	48.1 - 75.0	12.1 - 14.2	2.04 - 2.88	6.7 - 6.8
	$\overline{X}$ ± SD†	62.0 ± 7.1	13.1 ± 0.6	2.56 ± 0.25	6.7 ± 0.1
	CV, %¶	11.4	4.6	9.8	--
	CI#	56.9 - 67.1	12.7 - 13.5	2.38 - 2.74	--
Plot II	Range	47.3 - 68.9	14.2 - 17.6	3.27 - 3.99	6.7 - 6.9
	$\overline{X}$ ± SD†	57.0 ± 7.2	16.2 ± 1.2	3.62 ± 0.26	6.8 ± 0.1
	CV, %	12.6	7.4	7.2	--
	CI	51.8 - 62.2	15.3 - 17.1	3.43 - 3.81	--

*MFS = Moisture-free soil.

†$\overline{X}$ ± SD = Mean ± standard deviation, n = 10.

¶CV, % = Coefficient of variance, percent.

#CI = 95% confidence interval estimate, $t_{0.05}$.

TABLE C-2. SUMMARY OF THE SOIL ANALYSIS - NOVEMBER 1983.

		Oil and Grease (g/kg MFS*)	Volatile Material (% MFS*)	Total Kjeldahl Nitrogen (g/kg MFS*)	pH	Moisture Content (%WB†)
Plot I	Range	31.1 - 50.8	11.8 - 14.2	2.48 - 2.86	6.9	28.0 - 32.8
	$\overline{X}$ ± SD†	43.3 ± 5.6	13.1 ± 0.7	2.71 ± 0.13	6.9	31.0 ± 1.5
	CV, %#	12.9	5.3	4.8	—	4.8
	CI§	39.3 - 47.3	12.6 - 13.6	2.62 - 2.80	—	29.9 - 32.1
Plot II	Range	36.1 - 64.6	14.3 - 18.4	3.37 - 4.00	6.9 - 7.0	22.6 - 34.1
	$\overline{X}$ ± SD	48.7 ± 7.8	16.1 ± 1.2	3.67 ± 0.22	7.0	28.4 ± 3.9
	CV, %	16.0	7.4	6.0	—	13.7
	CI	43.1 - 54.3	15.2 - 17.0	3.51 - 3.83	—	25.6 - 31.2

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*MFS = Moisture-free soil.

†WB = Wet basis.

¶$\overline{X}$ ± SD = Mean ± standard deviation, n = 10.

#CV, % = Coefficient of variance, percent.

§CI = 95% confidence interval estimate, $t_{0.05}$.

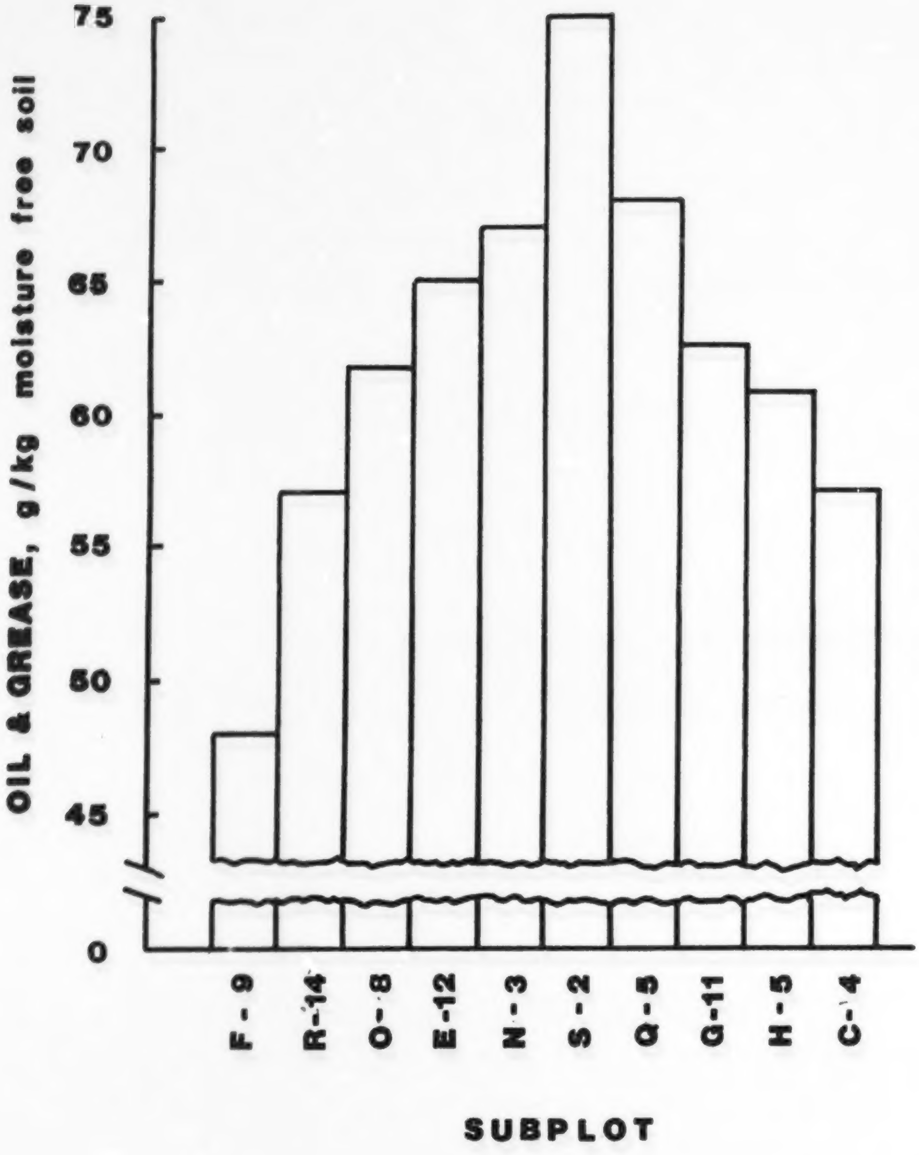

FIGURE C-1
DISTRIBUTION OF OIL AND GREASE IN THE SOIL OF PLOT I, JUNE 1983

TABLE C-3. RESULTS OF ANALYSES OF VARIANCE* FOR DIFFERENCES AMONG SUBPLOTS.

	Variance Ratio (F)		
	Oil and Grease	Total Kjeldahl Nitrogen	Volatile Material
June 1983			
Plot I	27.84	26.16	6.40
Plot II	44.74	9.54	21.24
November 1983			
Plot I	140.58	4.93	28.90
Plot II	153.34	12.45	126.95

*Subplots differ significantly ($P<0.05$) if $F>3.13$.

TABLE C-4. RESULTS OF ONE-WAY ANALYSES OF VARIANCE TO EVALUATE DIFFERENCES BETWEEN PLOTS AND BETWEEN SAMPLING DATES FOR EACH PLOT - MEAN VALUES.

	Oil and Grease (mg/g MFS*)	Total Kjeldahl Nitrogen, (mg/g MFS)	Volatile Material (% MFS)
June 1983			
Plot I	62.0 a†	2.56 c	13.1 e
Plot II	57.0 a	3.62 d	16.2 f
November 1983			
Plot I	43.3 b	2.71 c	13.1 e
Plot II	48.7 b	3.67 d	16.1 f

*MFS - Moisture-free soil.

†Means in the same column with a common letter are not significantly different ($P<0.05$) ($n=10$).

The differences noted in Table C-4 for the specific parameters can be explained in terms of the amount of material that was added to the soil at each plot and by the natural variations between plots. Table C-5 identifies the background characteristics as determined from control plots.

TABLE C-5. OILY WASTE LAND TREATMENT SITE - BACKGROUND SOIL CHARACTERISTICS* (DATA FROM CONTROL PLOTS).

Date	Oil & Grease (g/kg MFS)	Volatile Material (% MFS)	TKN (g/kg MFS)	pH
6/7/83	0.6 ± 0.2	8.8 ± 1.5	3.3 ± 0.7	5.8 ± 0.3
10/25/83	0.5 ± 0.1	9.4 ± 1.5	3.5 ± 0.8	5.8 ± 0.3

*n=4.

As a result of the waste application, the oil and grease and volatile material concentrations in the ZOI (Tables C-1 and C-2) were considerably greater than the background (Table C-5). In contrast, the nitrogen additions were small, compared to the amount in the control plot soils, and did not increase the ZOI concentrations measurably.

The results demonstrated that the spatial variation in industrial waste and treatment site ZOI characteristics can be statistically significant even when efforts are taken to insure uniform waste distribution and incorporation. The coefficient of variation of the oil and grease concentrations ranged from 11.4 to 16%, of the volatile material concentrations ranged from 4.6 to 7.4%, of the TKN concentrations ranged from 4.8 to 9.8% and of the moisture content concentrations ranged from 4.8 to 13.7% (Tables C-1 and C-2). At an actual land treatment site, where less care to obtain uniform distribution and mixing may be exerted, the coefficient of variation may be greater.

The spatial variation at a land treatment site is a function of site specific factors such as soil characteristics and methods of waste application and incorporation. Determination of specific site spatial variation is desirable as a prerequisite for identifying sampling requirements.

The data help determine the number of random samples needed to obtain a statistically significant estimate. Assuming that there will be random sampling from a large number of possible subplots within a land treatment site, the number of samples required to provide a desired level of confidence that the sample mean ($\overline{X}$) does differ from the average ZOI characteristics by more than an identified acceptable error can be calculated using the following relationship (8).

$$n = Z^2S^2/L^2 \qquad (1)$$

Where n = the required number of samples

Z = the normal deviate

S^2 = the population variance

L = the acceptable error

To use this relationship, it is first necessary to decide how accurate the sample estimate should be, i.e., to identify the limits of error (±L) that are acceptable. The required sample size is inversely proportional to the square of the acceptable error. The number of samples should provide an estimate that is useful, but the number should not be so large that the cost of sampling and analysis is excessive. The reason for obtaining the estimate of average site characteristics and the use of the results should be clearly identified before deciding how accurate the sample estimate should be.

If it is desired to express the acceptable error as a percentage of the sample mean, such as 30 mg/g ± 10%, the expected sample mean must be estimated before the required sample size can be calculated. The allowable error is then a function of the expected mean value and the required sample size and the accuracy of the estimate increases as the expected mean value decreases.

It can not be guaranteed that the sample mean ($\overline{X}$) will fall within the limits of acceptable error (±L) since the normal distribution curve extends from minus infinity to plus infinity. However, the probability that this will occur can be specified. Assuming that a 5% chance that the acceptable error (L) will be exceeded is acceptable, the value of the normal deviate (Z) is 1.96. For a 1% chance, the value of Z is 2.58. Values of Z for other probabilities can be obtained from appropriate statistical tables.

Finally, an estimate of the population variance (the standard deviation squared) is needed. It can be necessary to rely on previous experience or results from similar industrial waste land treatment sites as the basis for the estimate. If such information is not available, an educated guess may be necessary.

The results of this study indicate that spatial variations in ZOI characteristics vary little with time after waste is applied (Tables C-1 and C-2). Thus, the results from an initial estimate of average site characteristics should permit a more precise determination of sampling requirements for subsequent estimates.

An example can indicate how the number of samples can be determined. Using data from Plot I, June 1983 (Table 8), Table C-6 identifies the number of samples that are needed for different levels of acceptable error (±L) and different probabilities that these limits will be exceeded. A large number of samples is required for a small acceptable error and for small probabilities that the limits will be exceeded.

TABLE C-6. ILLUSTRATION OF THE NUMBER OF RANDOM SAMPLES REQUIRED FOR VARIOUS LEVELS OF ACCEPTABLE ERROR AND PROBABILITY.

Parameter	Acceptable Error	Number of Samples		
		Probability Error will be Exceeded		
	%*	10%**	5%**	1%**
Oil & Grease	20	1	2	3
(62.0 ± 7.1)†	10	4	5	9
	5	14	20	35
Total Kjeldahl	20	1	1	2
Nitrogen	10	3	4	7
(2.56 ± 0.25)†	5	11	15	26
Volatile Material	20	1	1	1
(13.1 ± 0.6)†	10	1	1	2
	5	3	4	6

*Percentage of sample mean that is the acceptable error.
**Probability that the acceptable error will be exceeded.
†Mean ± standard deviation for Plot I, June 1983 (Table C-1).

The data in Table C-6 provide an estimate of the error associated with samples taken from each of the plots to determine the changes in the characteristics of the plots during this study.

APPENDIX D

ANALYTICAL METHOD FOR THE DETERMINATION OF OIL AND GREASE IN SOIL SAMPLES

Introduction

This study was undertaken to determine the precision and accuracy of this method when used with soil samples. This information was of particular interest because oil and grease data were key analytical parameters in this study.

Analytical methods for oil and grease are based on the fact that oil and grease are insoluble in water, but are soluble in organic solvents. Usually, the sample is acidified to convert soaps to fatty acids before the oil and grease is extracted. In the determination of oil and grease, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in the solvent that is used. Unlike some elements or compounds, oils and greases are defined by the method used for their determination.

The solvents that have been used to determine oil and grease include petroleum ether, hexane, benzene, chloroform and methanol, or carbon tetrachloride. The solvent now commonly used in the water pollution control field is trichlorotrifluorethane (3). This solvent represents less of a hazard in the laboratory than many of the solvents noted above since trichlorotrifluoroethane is not flammable or explosive and has no known toxic properties (10).

Because no standard method exists for determining the oil and grease content of contaminated soils, the precision and accuracy of a modified form of the Soxhlet extraction method for sludge samples (Method 503D) (3), was evaluated to determine the use of this method for the oil and grease content of contaminated soils.

The results obtained by this method are empirical, and duplicate results can be obtained only by strict adherence to all details. The rate and time of extraction in the Soxhlet apparatus must be exactly as directed because of the varying solubilities of different materials. In addition, the length of time required to evaporate the solvent and cool the extracted material cannot be varied. There may be a gradual increase in weight, presumably due to absorption of oxygen or a gradual loss of weight due to volatilization (3). Compounds volatilized at or below the temperature of solvent evaporation will be lost during the evaporating process (3).

Soil samples containing known amounts, about 5% on a dry weight basis, of several types of oil were analyzed. For each oil-soil mixture, eight samples were analyzed. Eight samples of the soil without the oils were analyzed as blanks. In addition, a second series of five oil-soil mixtures were analyzed. These mixtures contained one to five percent, on a dry weight basis, of the oily waste applied to the field plots. For each oil concentration, two samples were analyzed.

Each stock mixture of oil and soil was prepared by adding the amount of oil, or oily waste, on a weight basis, needed to produce the desired concentration of oil or oily waste in 300 g of soil. After thorough mixing, these stock mixtures were stored at room temperatures (20 to 22°C) in screw-cap glass bottles until analyzed. When each oil-soil mixture was prepared, the dry matter content of the soil was determined so that the oil content of the stock mixtures could be expressed on a mg oil/g moisture free soil basis. Coefficients of variation for dry matter determinations never exceeded one percent.

The soil used for this study was from a site adjacent to the field plots. To eliminate soil characteristics as a variable, all of the soil used in this study was acquired at the beginning of the study and processed in the following manner. First, the soil was hand sorted to remove stones and vegetative matter. It then was mixed thoroughly for about 30 minutes using a portable concrete mixer and allowed to air dry at room temperature (20°C) until it was friable. When dry, the soil was pulverized and passed through a No. 16 U.S. Standard Sieve which had a mesh opening of 1.19 mm and stored at room temperature in a closed container until used.

The material used in this study were vegetable oil (partially hydrogenated soybean oil), No. 2 fuel oil, No. 6 fuel oil, SAE 30 non-detergent motor oil, and the oily waste used in the study.

Analytical Method

The method used in this study to determine the oil and grease content of soils and oil-soil mixtures was a modified form of the oil and grease extraction method for sludge samples (3). The following outlines the steps that were followed.

1. About 15 g of the soil or oil-soil mixture to be analyzed was placed in a preweighed, porcelain evaporating dish, 120 ml or larger, that contained a glass stirring rod. The dish with the glass rod and the soil was weighed again to determine the weight of the added soil or mixture.

2. Any soluble metallic soaps present were hydrolyzed by acidification with concentrated hydrochloric acid to pH 2.0 or lower using pH test paper as the method of measurement. To facilitate acidification,

distilled water to produce a smooth paste-like mixture was added before the acid.

3. After the acidified soil was mixed thoroughly with the stirring rod, the soil was dried by adding $MgSO_4 \cdot H_2O$ that was prepared by drying $MgSO_4 \cdot 7H_2O$ overnight at 150°C. Generally, 15-20 gm of $MgSO_4 \cdot H_2O$ was sufficient.

4. The acidified soil and $MgSO_4 \cdot H_2O$ were mixed using the stirring rod, and then was placed in a desiccator and allowed to cool.

5. After weighing the dish with its contents and the stirring rod, the contents of the dish were transferred to a porcelain mortar and ground.

6. A subsample of the material from step 5, containing no more than 150 mg of oil, was placed in a tared cellulose extraction thimble and weighed. The thimble was filled with small glass beads and placed in a Soxhlet extraction tube.

7. Using a preweighed extraction flask that had been dried at 103°C and cooled in a desiccator, the oil in the sample was extracted with 75 ml of reagent grade 1,1,2-trichloro-1,2,2-trifluoroethane for four hours at a rate of 20 cycles/hour.

8. After extraction, the extraction flask was placed in a 70°C water bath for one hour to evaporate the solvent. At the end of the hour, air was drawn through the flask for one minute using a vacuum pump to remove any remaining vapors.

9. Finally, the flask was weighed again after cooling in a desiccator for 30 minutes to determine the weight of extracted oil and grease.

Although this procedure is almost identical to method 503D for sludge samples outlined in (3), it differed in one important aspect. The Standard Methods procedure requires the quantitative transfer of the mixture of acidified sample and $MgSO_4 \cdot H_2O$ first to a mortar for grinding and then to an extraction thimble. The procedure used in this study eliminated possible errors associated with these transfers. It also permitted the use of a larger, more representative original sample which is an advantage when the oil and grease concentration limits the size of the sample that can be extracted. The subsample extracted contained portions of the original soil and the water and chemicals that were added.

The concentration of oil and grease in each soil sample was expressed as mg oil and grease per gram moisture free soil. The calculations were as follows:

$$\frac{\text{Oil and grease (mg/g)}}{\text{moisture free soil}} = \frac{\text{extraction flask weight gain (mg)}}{\text{soil extracted (g)} \times \text{dry matter fraction}} \qquad (2)$$

where:

$$\text{soil extracted(g)} = \text{subsample extracted(g)} \times \frac{\text{soil sample (g)}}{\text{soil sample} + H_2O + HCl + MgSO_4 \cdot H_2O\ \text{(g)}} \quad (3)$$

Results

The results of this evaluation are summarized in Tables D-1, D-2 and D-3. The precision (coefficient of variation) of this method was excellent (Tables D-1 and D-2). The accuracy (percent recovery) varied with type of oil (Table D-3) and with the concentration of the oily waste (Table 15).

Precision - As shown in Tables D-1 and D-2, the coefficient of variation of the oil-soil mixtures analyzed never exceeded four percent. There was little variation in precision as the concentration of oily waste varied (Table D-2).

The reason for the observed variability in the results for the blank, air dried soil samples is unclear, but it has been observed by the authors in other investigations. The coefficient of variation (50%) is large only in relative terms because the oil content of these samples was so low.

Routine analysis of the residue content of the reagent grade trichlorotrifluoroethane used in this study indicated that solvent contamination was not responsible for the observed variability. Residue after evaporation never exceeded 0.1 mg/100 ml.

Accuracy - The accuracy of this method varied with the oil type (Table D-3). With the exception of motor oil, the quantities added were underestimated.

The reasons for these differences in accuracy were not determined. It is known that extraction efficiency varies with oil composition and with the constituents being extracted. Only those substances that are soluble in the solvent used for extraction can be determined quantitatively (3).

The percent recovery for No. 2 fuel oil was the lowest among all of the oils used in this study (Table D-3). It is possible that losses of volatile constituents could have occurred during the preparation of the stock oil-soil mixtures as well as during sample acidification and drying. McGill and Rowell (11) have reported recoveries of about 70 percent for untopped crude oils (volatiles not removed) versus about 95 percent recovery for crude oils that had been topped at 21°C for 3 days. The solvent used was methylene chloride.

As shown in Table D-2, percent recovery increased as the concentration of oil in the oily waste-soil mixtures increased. Regression analysis of the analytical results indicated that the magnitude of the underestimate was constant. This indicates that the observed difference

TABLE D-1. PRECISION OF SOXHLET EXTRACTION WITH TRICHLOROTRIFLUOROETHANE TO MEASURE THE OIL AND GREASE CONTENT OF SOIL

	mg oil/g MFS*			
Oil	Range	$\bar{X}$ ± SD†	CI[¶]	CV,%[§]
Blank#	0.1 - 0.3	0.2 ± 0.1	0.1 - 0.3	50.0
Vegetable oil	41.1 - 45.8	43.2 ± 1.6	42.0 - 44.6	3.7
Fuel oil, No. 2	33.6 - 35.1	34.5 ± 0.6	34.0 - 35.0	1.7
Fuel oil, No. 6	41.9 - 45.0	43.9 ± 0.9	43.1 - 44.7	2.0
Motor oil, SAE 30**	50.8 - 57.1	54.7 ± 2.2	52.9 - 56.7	4.0
Oily waste	47.0 - 53.1	50.2 ± 2.0	48.5 - 51.9	4.0

*MFS = moisture-free soil
†$\bar{X}$ ± SD = mean ± standard deviation, n = 8.
¶CI = 95% confidence interval estimate, $t_{0.05}$
§CV, % = coefficient of variance, percent.
#air-dried soil
**non-detergent oil

TABLE D-2. PRECISION AND ACCURACY AS FUNCTIONS OF CONCENTRATION: FOR OILY WASTE-SOIL MIXTURES

mg oil/g MFS*			
Stock Mixture	Recovered	Coefficient of Variance (%)	Recovered (%)
10.5	8.8 ± 0.1†	1.1	83.8
21.0	18.0 ± 0.4	2.2	85.7
31.4	29.4 ± 0.1	0.3	93.6
41.9	40.5 ± 0.6	1.5	96.7
52.2	50.0 ± 0.6	1.2	95.8

*MFS = moisture-free soil
†blank corrected mean ± standard deviation, n = 2.

TABLE D-3. ACCURACY OF MEASURING THE OIL CONTENT OF SOIL — SOXHLET EXTRACTION WITH TRICHLOROTRIFLUOROETHANE

Oil	mg oil/g MFS*		Percent Recovered
	Stock Mixture	Recovered†	
Blank#	0	0.2 ± 0.1	-
Vegetable oil	50.6	43.0 ± 1.6	85.4
Fuel oil, No. 2	50.7	34.3 ± 0.6	68.0
Fuel oil, No. 6	52.9	43.7 ± 0.9	83.0
Motor oil, SAE 30**	53.5	54.5 ± 2.2	102.2
Oily waste	52.2	50.0 ± 2.0	96.2

*MFS = moisture-free soil
† = blank corrected mean ± standard deviation, n = 8.
# = air-dried soil
**non-detergent oil

TABLE D-4. SUMMARY OF RESULTS — MEASURING THE OIL CONTENT OF SOIL USING SOXHLET EXTRACTION WITH TRICHLOROTRIFLUOROETHANE

Oil	Coefficient of Variation (%)	Percent Recovered
Blank*	50.0	-
Vegetable oil	3.7	85.4
Fuel oil, No. 2	1.7	68.0
Fuel oil, No. 6	2.0	83.0
Motor oil, SAE 30†	4.0	102.2
Oily waste	4.0	96.2

*air-dried soil
†non-detergent oil

was not a function of concentration but of an unknown constant error. The difference between the intercept of the regression line (-2.24 mg oil and grease/gram moisture free soil) and the origin was tested statistically and was not found to be significant ($t<0.05$).

The results are summarized in Table D-4 and indicate that the modified method identified in this paper, which consists of acidification followed by Soxhlet extraction with trichlorotrifluoroethane, is precise and results in reasonable recoveries when used to measure the oil and grease content of contaminated soils.

APPENDIX E
CATION EXCHANGE CAPACITY OF THE SOIL IN THE FIELD PLOTS - 1982 to 1984 (n = 4)

TYPE OF APPLICATION	June 4 1982**	June 21 1982	October 5 1982	November 9 1982	June 21 1983	October 25 1983	April 26 1984
Natural Control	25.2 ± 8.5	26.3 ± 4.5	24.9 ± 3.6	23.9 ± 3.8	-*	24.8 ± 4.7	24.1 ± 3.9
Rototilled Control	25.0 ± 7.8	28.6 ± 7.0	25.3 ± 4.6	25.2 ± 5.3	-	-	-
Low	25.4 ± 3.0	26.4 ± 6.1	25.3 ± 2.9	22.8 ± 7.9	-	-	-
Medium	24.8 ± 2.9	24.5 ± 4.8	24.6 ± 2.3	24.3 ± 3.7	-	-	-
High	28.7 ± 7.5	27.5 ± 5.1	26.1 ± 4.8	23.5 ± 3.6	-	26.6 ± 4.8	23.1 ± 4.6
Very High+	-	-	-	-	25.5 ± 6.4	25.1 ± 5.8	22.9 ± 4.6

[*] Symbol indicates that the parameter was not measured on the noted dates in the respective plots.

[+] In June 1983, the low application plots became the very high application lots (see text SECTION 5).

[**] Background data before any wastes were applied or the plots were rototilled.

TABLE F-1. SUMMARY OF AVERAGE DATA FOR THE NATURAL CONTROL PLOTS (n = 4)

Sampling Date	pH	Soil Moisture (% WB)	Volatile Matter (% Soil MFB)	Oil and Grease (g/kg MFB)	TKN (g/kg MFB)	Soil Temperature (°C)
1982						
June 4**	5.9±0.1	-*	8.30±1.20	-	3.12±0.41	-
June 21	5.6±0.1	29.82±3.82	9.18±1.04	-	3.30±0.56	16.9±0.2
July 13	5.6±0.1	20.04±3.06	8.57±1.43	-	3.24±0.54	21.8±0.1
August 17	5.7±0.2	23.48±1.63	8.63±1.51	-	3.23±0.52	19.5±0.6
September 13	5.4±0.1	13.59±3.09	9.36±1.30	-	3.26±0.55	16.0±0.0
October 5	5.6±0.1	23.18±2.23	9.11±0.97	-	3.54±0.55	12.8±0.5
November 9	5.4±0.1	26.55±1.99	9.04±1.17	-	3.34±0.45	7.8±0.5
1983						
March 14	5.8±0.2	29.55±3.84	10.23±1.16	-	3.45±0.49	3.5±0.6
May 5	5.8±0.2	29.54±4.26	9.29±1.47	-	3.32±0.53	10.2±0.5
June 6	5.5±0.1	28.35±1.59	9.13±0.93	-	3.45±0.39	15.8±0.5
June 23	-	-	-	-	-	24.2±1.5
July 6	5.4±0.2	21.17±1.98	8.80±1.22	-	3.33±0.49	20.2±0.5
August 3	5.6±0.1	18.48±3.50	9.17±1.28	-	3.34±0.35	19.8±0.3
September 13	5.8±0.1	12.37±1.83	8.98±0.97	-	3.38±0.44	20.1±0.2
October 25	5.8±0.2	24.10±2.32	9.16±1.37	-	3.38±0.57	11.1±0.2
1984						
April 24	5.8±0.2	29.82±4.85	8.87±1.12	-	3.34±0.47	7.0±0.0
June 6	5.6±0.2	27.43±3.08	8.96±1.02	-	3.29±0.40	15.9±0.2
July 9	5.5±0.2	28.9±3.66	8.95±0.86	-	3.12±0.33	16.2±0.3

*Symbol means that the parameter was not measured on the noted dates.

**Background sample taken before any wastes were applied or the plots were rototilled.

TABLE F-2. SUMMARY OF AVERAGE DATA FOR THE ROTOTILLED CONTROL PLOTS (n = 4)

Sampling Date	pH	Soil Moisture (% WB)	Volatile Matter (% Soil MFB)	Oil and Grease (g/kg MFB)	TKN (g/kg MFB)	Soil Temperature (°C)
1982						
June 4**	5.8±0.1	-*	9.12±2.60	-	3.25±0.63	-
June 21	5.6±0.1	31.17±4.78	9.20±1.72	-	-	16.0±0.4
July 13	5.7±0.2	23.50±4.59	8.48±1.46	-	-	22.5±0.6
August 17	5.7±0.2	25.72±3.98	8.73±1.21	-	3.32±0.65	19.8±0.5
September 13	5.8±0.2	16.40±6.08	9.58±2.00	-	3.55±0.88	16.0±0.8
October 5	5.8±0.2	23.23±3.46	9.35±1.28	0.21±0.10	3.50±0.61	13.0±0.0
November 9	5.8±0.2	27.44±5.24	9.19±1.82	0.33±0.20	3.49±0.84	8.0±0.8
1983						
March 14	5.9±0.4	31.00±5.28	9.16±1.33	0.22±0.05	3.47±0.61	3.5±0.6
May 5	5.8±0.6	29.24±5.10	9.15±1.66	0.68±0.16	3.25±0.92	10.2±0.5
June 6	5.8±0.3	25.20±5.22	9.12±1.38	0.65±0.17	3.33±0.70	17.0±1.8
June 23	-	-	-	-	-	23.0±0.0
July 6	5.8±0.4	17.27±3.67	9.14±1.54	0.59±0.24	3.49±0.76	20.4±0.5
August 3	5.8±0.3	15.71±4.00	9.58±1.69	0.44±0.12	3.53±0.79	20.1±0.2
September 13	5.7±0.3	11.76±4.12	9.41±1.64	0.68±0.19	3.54±0.79	20.0±0.7
October 25	5.8±0.3	24.43±3.05	9.36±1.44	0.54±0.11	3.37±0.66	11.1±0.2
1984						
April 24	5.8±0.2	28.98±4.84	8.79±1.35	0.57±0.12	3.49±0.62	7.0±0.0
June 6	5.8±0.2	27.32±4.48	8.75±1.02	-	3.23±0.51	16.5±1.0
July 9	5.6±0.4	24.25±5.81	9.11±1.35	0.36±0.10	3.30±0.61	16.4±0.6

*Symbol means that the parameter was not measured on the noted dates.

**Background sample taken before any wastes were applied or the plots were rototilled.

TABLE F-3. SUMMARY OF AVERAGE DATA FOR THE LOW APPLICATION PLOTS (n = 4)

Sampling Date	pH	Soil Moisture (% WB)	Volatile Matter (% Soil MFB)	Oil and Grease (g/kg MFB)	TKN (g/kg MFB)	Soil Temperature (°C)
1982						
June 4**	6.0±0.2	-*	8.56±1.36	-	-	-
June 21	5.7±0.2	31.96±4.77	9.44±1.74	-	-	16.1±0.2
July 13	5.4±0.2	23.33±3.52	8.36±1.13	-	-	22.8±0.5
August 17	5.5±0.2	24.29±1.82	8.82±1.29	-	3.29±0.58	19.8±0.5
September 13	5.8±0.2	16.32±2.30	9.16±1.35	-	3.51±0.64	15.8±0.5
October 5	5.8±0.2	23.69±1.45	9.27±1.30	0.39±0.15	3.54±0.62	12.5±0.6
November 9	6.0±0.1	27.48±1.83	10.42±1.83	5.48±1.08	3.71±0.84	8.0±0.8
1983						
March 14	6.2±0.3	29.72±5.77	10.01±1.54	4.60±0.91	3.50±0.65	3.2±1.0
May 10	6.1±0.2	30.53±3.19	10.31±1.23	4.18±0.46	3.54±0.62	10.0±0.8
June 6	6.0±0.1	26.01±2.70	10.32±1.51	3.79±0.73	3.58±0.68	16.5±0.6

*Symbol indicates that the parameter was not measured on the noted dates.

**Background sample taken before any wastes were applied or the plots were rototilled.

TABLE F-4. SUMMARY OF AVERAGE DATA FOR THE MEDIUM APPLICATION PLOTS (n = 4)

Sampling Date	pH	Soil Moisture (% WB)	Volatile Matter (% Soil MFB)	Oil and Grease (g/kg MFB)	TKN (g/kg MFB)	Soil Temperature (°C)
1982						
June 4**	5.9±0.2	-*	8.22±1.17	-	-	-
June 21	5.5±0.2	30.38±4.86	8.90±1.02	0.94±0.11	3.20±0.56	16.0±0.0
July 13	5.5±0.2	23.80±2.80	8.44±0.98	-	-	22.8±0.5
August 17	5.4±0.3	24.71±2.12	8.68±0.88	-	3.08±0.48	19.8±0.5
September 13	5.8±0.2	14.94±2.86	9.06±0.96	0.54±0.17	3.40±0.51	16.0±0.0
October 5	5.8±0.2	22.76±1.12	8.86±0.74	0.66±0.09	3.47±0.49	13.0±0.0
November 9	6.0±0.2	27.00±1.84	10.41±1.60	9.31±2.92	3.30±0.66	8.2±0.5
1983						
March 14	6.3±0.2	31.04±4.85	10.00±1.53	8.64±2.93	3.36±0.65	3.5±0.6
May 5	6.2±0.1	28.28±1.45	10.26±1.15	8.82±1.58	3.28±0.41	10.2±1.0
June 6	6.2±0.3	26.49±1.47	10.49±0.78	9.24±2.24	3.22±0.42	16.5±0.6
June 23	-	-	-	-	-	24.8±0.5
July 6	6.3±0.1	19.65±1.59	10.16±1.15	7.30±1.78	3.44±0.64	21.4±0.5
August 3	6.1±0.1	18.78±1.74	9.92±1.22	6.88±1.04	3.44±0.66	19.9±0.2
September 13	6.1±0.1	12.76±2.13	10.27±1.44	7.04±1.94	3.50±0.59	20.2±0.3
October 25	6.2±0.1	25.19±1.97	10.32±1.07	6.01±0.47	3.41±0.55	11.0±0.0
1984						
April 24	6.2±0.1	29.06±2.90	9.32±0.56	4.98±1.25	3.28±0.34	7.2±0.3
June 6	6.1±0.1	27.89±3.21	10.02±0.94	5.01±0.38	3.40±0.48	16.2±1.0
July 9	6.2±0.1	29.26±4.87	9.22±0.56	4.37±1.08	3.17±0.34	16.9±0.2

*Symbol indicates that the parameter was not measured on the noted dates.

**Background sample taken before any wastes were applied or the plots were rototilled.

TABLE F-5. SUMMARY OF AVERAGE DATA FOR THE HIGH APPLICATION PLOTS (n = 4)

Sampling Date	pH	Soil Moisture (% WB)	Volatile Matter (% Soil MFB)	Oil and Grease (g/kg MFB)	TKN (g/kg MFB)	Soil Temperature (°C)
1982						
June 4**	5.8±0.2	-*	8.83±1.34	-	3.14±0.57	-
June 21	5.6±0.2	30.10±4.31	9.21±1.33	1.98±0.38	3.42±0.54	16.1±0.6
July 13	5.7±0.1	25.68±2.24	8.98±1.06	1.08±0.28	-	22.8±0.5
August 17	5.8±0.2	26.79±3.25	9.33±1.59	1.08±0.31	3.08±0.50	19.8±0.5
September 13	5.8±0.2	17.10±4.84	10.37±1.24	1.12±0.24	3.72±0.57	16.2±0.5
October 5	5.9±0.2	24.11±3.54	9.94±1.35	1.20±0.27	3.74±0.59	13.5±0.6
November 9	6.2±0.2	26.90±3.28	11.80±1.81	19.60±2.83	3.54±0.55	8.2±0.5
1983						
March 14	6.4±0.1	30.01±4.55	11.74±1.50	17.90±5.12	3.60±0.49	3.2±1.0
May 5	6.4±0.1	31.77±7.30	11.74±1.69	14.86±6.40	3.61±0.81	10.0±0.0
June 6	6.2±0.1	27.20±3.32	11.20±1.50	14.86±3.17	3.50±0.47	17.0±1.4
June 23	-	-	-	-	-	24.5±0.6
July 6	6.5±0.1	22.13±1.99	11.51±1.44	15.99±1.20	3.62±0.52	22.0±0.0
August 3	6.5±0.2	20.37±2.98	11.14±1.47	10.83±1.85	3.42±0.67	20.5±0.7
September 13	6.4±0.1	14.58±2.44	11.89±1.13	13.42±2.29	3.73±0.53	20.0±0.0
October 25	6.4±0.1	25.76±2.89	11.13±1.78	9.40±2.35	3.34±0.54	11.4±0.2
1984						
April 24	6.4±0.1	29.36±4.20	11.26±1.15	10.35±2.16	3.65±0.49	7.0±0.0
June 6	6.2±0.1	27.77±3.18	10.44±1.06	7.78±2.34	3.44±0.44	16.5±0.6
July 9	6.5±0.1	27.88±4.70	10.87±1.18	6.44±0.98	3.50±0.56	16.9±0.8

*Symbol indicates that the parameter was not measured on the noted dates.

**Background sample taken before any wastes were applied or the plots were rototilled.

TABLE F-6. SUMMARY OF AVERAGE DATA FOR THE VERY HIGH APPLICATION PLOTS (n = 4)

Sampling Date	pH	Soil Moisture (% WB)	Volatile Matter (% Soil MFB)	Oil and Grease (g/kg MFB)	TKN (g/kg MFB)	Soil Temperature (°C)
1983						
June 23	7.1±0.2	22.64±1.92	14.98±1.33	56.16±2.09	3.24±0.66	26.2±0.5
July 6	6.9±0.1	20.26±1.59	14.24±1.97	55.85±6.52	3.22±0.61	25.6±1.1
August 3	6.8±0.0	19.46±3.09	15.28±1.75	51.10±2.59	3.42±0.52	24.1±1.6
September 13	6.7±0.1	20.96±2.00	14.20±1.34	37.86±4.93	3.61±0.62	22.9±0.2
October 25	6.8±0.1	28.56±1.92	13.86±1.36	34.22±1.12	3.31±0.66	12.4±0.9
1984						
April 24	6.7±0.1	31.94±3.94	13.90±1.64	31.63±3.73	3.56±0.66	7.3±0.3
June 6	6.8±0.0	31.56±1.84	13.56±1.22	31.72±2.46	3.45±0.62	19.6±1.8
July 9	6.7±0.1	30.92±1.21	12.67±1.73	28.63±3.58	3.50±0.65	19.1±0.6

TABLE G-1
METAL CONCENTRATIONS⁺ IN THE SOILS OF NATURAL CONTROL PLOTS
(mg/kg MOISTURE FREE SOIL) (n = 4)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum	27,200	25,300	22,800	23,700	26,000	23,900	26,300
(Al)	±2,300	±2,400	±1,700	±4,000	±3,900	±2,500	±2,400
Cadmium	<**	<	<	<	<	<	<
(Cd)							
Calcium	3,200	3,300	3,200	3,200	3,300	3,200	3,400
(Ca)	±720	±700	±800	±700	±710	±800	±700
Chromium	31	29	26	29	30	27	30
(Cr)	±3	±3	±2	±5	±4	±3	±3
Copper	14	14	16	17	16	16	15
(Cu)	±3	±3	±4	±4	±3	+4	±3
Iron	26,700	26,300	27,000	27,300	28,000	27,000	27,400
(Fe)	±2,000	±2,300	±3,000	±2,900	±2,300	±2,300	±2,500
Lead	<	<	24	25	25	19	25
(Pb)			±4	±3	±1	±2	±8
Magnesium	4,800	4,800	4,400	4,500	4,600	4,400	4,500
(Mg)	±290	±330	±370	±350	±330	±360	±360
Manganese	650	590	540	570	580	530	600
(Mn)	±210	±140	±200	±200	±86	±140	±140
Nickel	27	26	24	26	25	25	26
(Ni)	±2	±3	±4	±3	±2	±3	±3
Potassium	3,700	3,100	1,900	2,000	3,000	2,400	3,000
(K)	±630	±650	±350	±700	±830	±660	±700
Sodium	220	190	470	420	120	78	<
(Na)	±57	±34	±60	±35	±23	±21	
Titanium	160	110	53	74	100	61	86
(Ti)	±48	±14	±25	±36	±25	±21	±29
Vanadium	43	38	32	37	40	36	40
(V)	±4	±4	±3	±7	±6	±5	±4
Zinc	98	99	93	99	98	92	105
(Zn)	±13	±14	±11	±15	±15	±11	±17

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

**< = less than the limit of detection determined by instrument sensitivity, sample dilution, and matrix interference.

TABLE G-2
METAL CONCENTRATIONS+ IN THE SOILS OF ROTOTILLED CONTROL PLOTS
(mg/kg MOISTURE FREE SOIL)(n = 4)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum	27,900	27,200	22,200	21,400	28,000	24,000	25,800
(Al)	±3,000	±3,900	±3,400	±1,700	±4,400	±860	±3,700
Cadmium	<**	<	<	<	<	<	<
(Cd)							
Calcium	3,400	3,500	3,500	3,500	3,500	3,400	3,500
(Ca)	±1,000	±1,100	±1,100	±1,200	±1,000	±1,000	±1,100
Chromium	31	30	27	25	31	27	29
(Cr)	±3	±5	±4	±6	±5	±1	±4
Copper	15	15	18	20	18	15	15
(Cu)	±3	±3	±3	±4	±4	±3	±3
Iron	27,500	26,700	27,700	26,400	28,000	27,000	27,100
(Fe)	±2,900	±1,800	±2,900	±2,300	±2,500	±2,000	±2,800
Lead	<	<	23	25	24	19	22
(Pb)			±3	±2	±2	±2	±2
Magnesium	4,800	4,800	4,300	4,200	4,800	4,400	4,480
(Mg)	±280	±380	±520	±380	±440	±240	±460
Manganese	630	600	600	580	560	570	600
(Mn)	±220	±230	±280	±240	±190	±250	±270
Nickel	26	27	24	25	25	24	25
(Ni)	±2	±3	±4	±2	±3	±3	±3
Potassium	3,500	3,800	1,700	1,600	3,600	2,300	2,800
(K)	±820	±1,100	±690	±240	±1,100	±330	±890
Sodium	210	230	460	330	140	83	40
(Na)	±47	±36	±190	±80	±43	±4	±6
Titanium	130	120	27	64	130	64	75
(Ti)	±24	±30	±11	±28	±38	±24	±18
Vanadium	41	40	31	34	43	36	39
(V)	±5	±7	±5	±3	±7	±2	±6
Zinc	98	98	93	96	98	88	95
(Zn)	±14	±12	±13	±17	±15	±10	±22

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

**<= less than the limit of detection determined by instrument sensitivity, sample dilution, and matrix interference.

TABLE G-3
METAL CONCENTRATIONS+ IN THE SOIL OF THE LOW APPLICATION PLOTS (mg/kg MOISTURE FREE SOIL)(n = 4)

	Sampling Date				
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83
Aluminum (Al)	25,800 ±2,700	27,000 ±2,800	23,700 ±2,500	25,000 ±2,400	25,000 ±11,000
Cadmium (Cd)	<**	<	<	<	<
Calcium (Ca)	3,300 ±650	3,500 ±780	3,450 ±700	4,800 ±1,000	3,800 ±1,800
Chromium (Cr)	29 ±3	32 ±3	29 ±2	48 ±4	41 ±18
Copper (Cu)	14 ±2	15 ±2	17 ±2	21 ±3	15 ±7
Iron (Fe)	26,300 ±2,600	26,000 ±3,000	26,700 ±1,700	26,200 ±3,000	22,000 ±9,300
Lead (Pb)	<	27 ±2	23 ±2	31 ±3	24 ±9
Magnesium (Mg)	4,700 ±400	4,800 ±430	4,300 ±250	4,400 ±420	3,900 ±1,700
Manganese (Mn)	610 ±100	650 ±120	530 ±60	560 ±100	440 ±180
Nickel (Ni)	26 ±2	26 ±3	24 ±2	25 ±3	20 ±9
Potassium (K)	3,200 ±860	3,800 ±850	2,300 ±550	2,500 ±750	3,600 ±1,500
Sodium (Na)	210 ±46	220 ±46	530 ±130	440 ±25	150 ±60
Titanium (Ti)	120 ±15	120 ±15	52 ±27	97 ±22	140 ±55
Vanadium (V)	38 ±4	40 ±4	34 ±3	40 ±3	38 ±16
Zinc (Zn)	100 ±5	105 ±11	100 ±13	130 ±13	110 ±51

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

**< = less than the limit of detection determined by instrument sensitivity, sample dilution, and matrix interference.

TABLE G-4
METAL CONCENTRATIONS+ IN THE SOIL OF MEDIUM APPLICATION PLOTS (mg/kg MOISTURE FREE SOIL)(n = 4)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum (Al)	26,900 ±3,000	26,100 ±890	22,000 ±2,000	25,600 ±1,800	31,000 ±2,900	26,300 ±3,100	31,000 ±2,400
Cadmium (Cd)	<**	<	<	<	<	<	<
Calcium (Ca)	3,300 ±770	3,600 ±620	3,300 ±660	5,000 ±1,200	5,600 ±520	4,500 ±1,800	5,400 ±860
Chromium (Cr)	30 ±3	33 ±1	29 ±2	51 ±8	62 ±4	43 ±20	57 ±5
Copper (Cu)	15 ±2.9	14 ±2.2	17 ±0.9	21 ±3.3	21 ±1.2	17 ±4.6	19 ±2.5
Iron (Fe)	25,900 ±1,900	25,700 ±1,600	25,500 ±1,000	26,600 ±2,500	27,000 ±1,600	26,400 ±2,000	27,300 ±3,000
Lead (Pb)	<	<	24 ±2	32 ±4	30 ±1	22 ±5	27 ±2
Magnesium (Mg)	4,700 ±360	4,700 ±220	4,100 ±160	4,500 ±400	4,800 ±310	4,400 ±370	4,700 ±400
Manganese (Mn)	570 ±130	570 ±140	500 ±120	530 ±110	530 ±110	520 ±140	620 ±190
Nickel (Ni)	25 ±3	26 ±2	22 ±2	26 ±3	25 ±2	24 ±3	26 ±3
Potassium (K)	3,700 ±970	3,400 ±400	1,900 ±430	2,700 ±810	4,900 ±1,000	3,200 ±1,400	4,700 ±1,000
Sodium (Na)	220 ±50	210 ±30	500 ±100	430 ±38	220 ±55	130 ±59	64 ±31
Titanium (Ti)	135 ±11	120 ±19	58 ±7	100 ±10	160 ±44	94 ±53	140 ±21
Vanadium (V)	41 ±4	38 ±2	32 ±3	40 ±3	47 ±5	40 ±6	48 ±4
Zinc (Zn)	96 ±7	104 ±7	94 ±10	140 ±19	160 ±7	120 ±40	150 ±15

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

**< = less than the limit of detection determined by instrument sensitivity, sample dilution, and matrix interference.

TABLE G-5
METAL CONCENTRATIONS⁺ IN THE SOIL OF HIGH APPLICATION PLOTS
(mg/kg MOISTURE FREE SOIL)(n = 4)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum (Al)	26,900 ±2,300	29,400 ±2,300	23,200 ±3,600	27,200 ±2,600	36,000 ±3,100	26,800 ±2,900	31,900 ±5,000
Cadmium (Cd)	<**	<	<	<	<	<	<
Calcium (Ca)	3,600 ±1,000	4,100 ±700	3,900 ±860	5,800 ±740	7,100 ±990	4,000 ±1,300	6,400 ±1,030
Chromium (Cr)	31 ±4	39 ±5	33 ±4	63 ±8	79 ±1	36 ±11	68 ±12
Copper (Cu)	16 ±3	16 ±2	18 ±4	24 ±1	25 ±3	18 ±4	22 ±4
Iron (Fe)	27,500 ±2,500	26,900 ±2,100	26,200 ±2,700	27,300 ±1,100	27,000 ±1,900	27,300 ±1,700	27,400 ±2,400
Lead (Pb)	<	<	25 ±4	42 ±15	34 ±2	22 ±4	30 ±3
Magnesium (Mg)	4,900 ±390	4,900 ±320	4,300 ±600	4,500 ±330	5,100 ±350	4,500 ±340	4,800 ±510
Manganese (Mn)	700 ±210	600 ±170	660 ±230	650 ±190	640 ±140	660 ±190	630 ±190
Nickel (Ni)	27 ±2	27 ±3	24 ±4	26 ±2	26 ±2	26 ±3	26 ±3
Potassium (K)	3,400 ±860	4,100 ±640	2,100 ±1,000	3,100 ±910	7,000 ±1,200	3,000 ±920	5,000 ±1,900
Sodium (Na)	200 ±41	240 ±17	500 ±130	600 ±140	300 ±32	120 ±28	67 ±55
Titanium (Ti)	140 ±50	150 ±26	66 ±33	120 ±47	240 ±43	96 ±31	140 ±59
Vanadium (V)	42 ±6	44 ±4	34 ±6	44 ±5	56 ±5	42 ±6	49 ±9
Zinc (Zn)	100 ±15	120 ±19	100 ±12	160 ±26	200 ±25	110 ±32	180 ±30

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

**< = less than the limit of detection determined by instrument sensitivity, sample dilution, and matrix interference.

TABLE G-6
METAL CONCENTRATIONS+ IN THE SOIL OF THE VERY HIGH APPLICATION PLOTS (mg/kg MOISTURE FREE SOIL)(n = 4)

	Sampling Date		
Metal	6/21/83	10/25/83	6/6/84
Aluminum (Al)	32,200 ±2,500	32,200 ±3,400	35,200 ±4,300
Cadmium (Cd)	<	<*	<
Calcium (Ca)	13,200 ±960	12,400 ±3,800	13,700 ±1,100
Chromium (Cr)	145 ±10	140 ±40	160 ±11
Copper (Cu)	43 ±3	35 ±9	38 ±4
Iron (Fe)	24,000 ±1,600	25,200 ±1,800	25,500 ±3,000
Lead (Pb)	53 ±3	48 ±12	54 ±3
Magnesium (Mg)	4,600 ±330	4,600 ±360	4,800 ±520
Manganese (Mn)	520 ±76	500 ±39	520 ±75
Nickel (Ni)	23 ±3	25 ±2	26 ±3
Potassium (K)	6,000 ±770	5,700 ±1,300	7,400 ±1,800
Sodium (Na)	370 ±43	310 ±62	210 ±68
Titanium (Ti)	220 ±16	200 ±52	220 ±40
Vanadium (V)	50 ±4	50 ±6	55 ±7
Zinc (Zn)	340 ±19	320 ±89	380 ±22

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* < = less than the limit of detection determined by instrument sensitivity, sample dilution, and matrix interference.

TABLE H-1
ORGANIC CONCENTRATIONS IN THE SOILS OF PLOT 5 - A VERY HIGH APPLICATION PLOT (mg/kg MOISTURE FREE SOIL)+

Organic Compound	Sampling Date 6/23/83	7/6/83	8/3/83	9/14/83	10/25/83
Napthalene	25	<9*	<10*	<10*	<10*
2-methyl-napthalene	45	21	"	"	"
1-methyl-napthalene	74	13	"	"	"
2,6-dimethyl-napthalene	329	46	"	"	"
1,3-dimethyl-napthalene	491	41	"	"	"
2,3-dimethyl-napthalene	159	57	"	"	"
1,2-dimethyl-napthalene	91	61	"	"	"
C_8 Alkane	<10*	<10	"	"	"
C_9 Alkane	<10	<10	"	"	"
C_{10} Alkane	21	<10	"	"	"
C_{11} Alkane	55	<10	"	"	"
C_{12} Alkane	91	16	"	"	"
C_{13} Alkane	127	29	"	"	"
C_{14} Alkane	203	54	"	"	"
C_{15} Alkane	193	104	"	9	"
C_{16} Alkane	265	122	"	<7*	"
C_{17} Alkane	235	134	"	9	"
C_{18} Alkane	216	140	6	14	"
C_{19} Alkane	199	156	10	11	"
C_{20} Alkane	172	156	8	13	"
C_{21} Alkane	118	108	<10	<10	"
C_{22} Alkane	79	72	"	"	"
C_{23} Alkane	61	46	"	"	"
C_{24} Alkane	55	48	"	"	"
C_{25} Alkane	129	59	"	"	"
C_{26} Alkane	44	38	"	"	"
Biphenyl	32	<10	"	"	"
3-methyl-biphenyl	64	"	"	"	"
Dibenzofuran	66	"	"	"	"
Fluorene	16	"	"	"	"
Phenanthrene	33	"	"	"	"
Anthracene	17	"	"	"	"
Carbazole	<10	"	"	"	"
1-methylphenanthrene	42	"	"	"	"
Pyrene	37	26	20	10	6

+ the very high waste application occurred on June 19,1983.

* less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE H-2
ORGANIC CONCENTRATIONS IN THE SOILS OF PLOT 12 - A VERY HIGH APPLICATION PLOT (mg/kg MOISTURE FREE SOIL)+

Organic Compound	Sampling Date				
	6/23/83	7/6/83	8/3/83	9/14/83	10/25/83
Napthalene	22	<10*	<10*	<10*	<10*
2-methyl-napthalene	37	17	"	"	"
1-methyl-napthalene	36	<10	"	"	"
2,6-dimethyl-napthalene	257	<10	"	"	"
1,3-dimethyl-napthalene	382	23	"	"	"
2,3-dimethyl-napthalene	123	24	"	"	"
1,2-dimethyl-napthalene	62	<10	"	"	"
C_8 Alkane	<10*	"	"	"	"
C_9 Alkane	<10	"	"	"	"
C_{10} Alkane	24	"	"	"	"
C_{11} Alkane	54	"	"	"	"
C_{12} Alkane	99	11	"	"	"
C_{13} Alkane	185	44	"	"	"
C_{14} Alkane	276	87	10	"	"
C_{15} Alkane	386	123	26	13	"
C_{16} Alkane	341	77	17	23	"
C_{17} Alkane	402	110	25	19	15
C_{18} Alkane	377	112	26	23	<10
C_{19} Alkane	344	120	23	18	"
C_{20} Alkane	295	126	35	25	"
C_{21} Alkane	188	80	17	16	"
C_{22} Alkane	135	58	14	<10	"
C_{23} Alkane	98	54	22	"	"
C_{24} Alkane	84	52	14	"	"
C_{25} Alkane	160	114	41	16	31
C_{26} Alkane	44	66	17	<10	<10
Biphenyl	31	24	<10	"	"
3-methyl-biphenyl	61	10	"	"	"
Dibenzofuran	66	23	"	"	"
Fluorene	22	23	"	"	"
Phenanthrene	23	<10	"	"	"
Anthracene	16	"	"	"	"
Carbazole	<10	"	"	"	"
1-methylphenanthrene	51	13	"	"	"
Pyrene	25	20	"	"	"

+ the very high waste application occurred on June 19,1983.

* less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE H-3
ORGANIC CONCENTRATIONS IN THE SOILS OF PLOT 18 - A VERY HIGH APPLICATION PLOT (mg/kg MOISTURE FREE SOIL)+

Organic Compound	Sampling Date 7/6/83	 10/25/83	 6/6/84
Napthalene	<10*	<7*	<5*
2-methyl-napthalene	15	"	"
1-methyl-napthalene	<10	"	"
2,6-dimethyl-napthalene	"	"	"
1,3-dimethyl-napthalene	"	"	"
2,3-dimethyl-napthalene	"	"	"
1,2-dimethyl-napthalene	17	"	"
C_8 Alkane	<10	"	"
C_9 Alkane	"	"	"
C_{10} Alkane	"	"	"
C_{11} Alkane	"	"	"
C_{12} Alkane	12	"	"
C_{13} Alkane	<10	"	"
C_{14} Alkane	18	"	"
C_{15} Alkane	36	7	"
C_{16} Alkane	14	<7	"
C_{17} Alkane	39	9	"
C_{18} Alkane	45	12	"
C_{19} Alkane	42	10	"
C_{20} Alkane	44	10	"
C_{21} Alkane	30	<7	"
C_{22} Alkane	23	"	"
C_{23} Alkane	16	8	"
C_{24} Alkane	18	<7	"
C_{25} Alkane	36	"	"
C_{26} Alkane	35	10	"
Biphenyl	13	<7	"
3-methyl-biphenyl	<10	"	"
Dibenzofuran	"	"	"
Fluorene	19	"	"
Phenanthrene	10	"	"
Anthracene	40	"	"
Carbazole	<10	"	"
1-methylphenanthrene	"	"	"
Pyrene	11	"	"

+ the very high waste application occurred on June 19,1983.

* less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE H-4
ORGANIC CONCENTRATIONS IN THE SOILS OF PLOT 14 - A HIGH APPLICATION PLOT (mg/kg MOISTURE FREE SOIL)$^+$

	Sampling Date			
Organic Compound	11/9/82	6/6/83	10/25/83	6/6/84
Napthalene	<8*	<10*	<6*	<6*
2-methyl-napthalene	"	"	"	"
1-methyl-napthalene	"	"	"	"
2,6-dimethyl-napthalene	"	"	"	"
1,3-dimethyl-napthalene	"	"	"	"
2,3-dimethyl-napthalene	"	"	"	"
1,2-dimethyl-napthalene	"	"	"	"
C_{8} Alkane	"	"	"	"
C_{9} Alkane	"	"	"	"
C_{10} Alkane	"	"	"	"
C_{11} Alkane	"	"	"	"
C_{12} Alkane	"	"	"	"
C_{13} Alkane	"	"	"	"
C_{14} Alkane	"	"	"	"
C_{15} Alkane	20	"	"	"
C_{16} Alkane	33	14	"	"
C_{17} Alkane	43	<10	"	"
C_{18} Alkane	51	14	"	"
C_{19} Alkane	48	10	"	"
C_{20} Alkane	40	10	"	"
C_{21} Alkane	27	<10	"	"
C_{22} Alkane	20	15	"	"
C_{23} Alkane	11	<10	"	"
C_{24} Alkane	10	"	"	"
C_{25} Alkane	15	"	"	"
C_{26} Alkane	8	"	"	"
Biphenyl	<8	"	"	"
3-methyl-biphenyl	"	"	"	"
Dibenzofuran	"	"	"	"
Fluorene	"	"	"	"
Phenanthrene	"	"	"	"
Anthracene	"	"	"	"
Carbazole	"	"	"	"
1-methylphenanthrene	"	"	"	"
Pyrene	"	"	"	"

+ the high waste application occurred on October 28, 1982.

* less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE H-5
ORGANIC CONCENTRATIONS IN THE SOILS OF PLOT 11 - A MEDIUM APPLICATION PLOT (mg/kg MOISTURE FREE SOIL)+

	Sampling Date	
Organic Compound	11/9/82	10/25/83
Napthalene	<5*	<5*
2-methyl-napthalene	"	"
1-methyl-napthalene	"	"
2,6-dimethyl-napthalene	"	"
1,3-dimethyl-napthalene	"	"
2,3-dimethyl-napthalene	"	"
1,2-dimethyl-napthalene	"	"
C_8 Alkane	"	"
C_9 Alkane	"	"
C_{10} Alkane	"	"
C_{11} Alkane	"	"
C_{12} Alkane	"	"
C_{13} Alkane	"	"
C_{14} Alkane	"	"
C_{15} Alkane	14	"
C_{16} Alkane	19	"
C_{17} Alkane	24	"
C_{18} Alkane	28	"
C_{19} Alkane	29	"
C_{20} Alkane	26	"
C_{21} Alkane	18	"
C_{22} Alkane	11	"
C_{23} Alkane	8	"
C_{24} Alkane	9	"
C_{25} Alkane	7	"
C_{26} Alkane	6	"
Biphenyl	<5	"
3-methyl-biphenyl	"	"
Dibenzofuran	"	"
Fluorene	"	"
Phenanthrene	"	"
Anthracene	"	"
Carbazole	"	"
1-methylphenanthrene	"	"
Pyrene	"	"

+ the medium application occurred on October 28, 1982.

* less than the noted detection limit of the analytical methods used when calculated in terms of these units.

TABLE I-1
AVERAGE EARTHWORM DENSITY FOUND IN THE FIELD PLOTS (number per m^2)

	Type of Application					
Date	Natural Controls	Rototilled Controls	Low	Medium	High	Very High
1982						
June 4[**]	334^{a}[*]	596^{a}	392^{a}	503^{a}	573^{a}	--[+]
June 21	467^{a}	259^{a}	191^{a}	332^{a}	200^{a}	--
July	204^{a}	119^{a}	48^{a}	85^{a}	71^{a}	--
August	255^{a}	78^{a}	137^{a}	156^{a}	89^{a}	--
September	63^{a}	89^{a}	59^{a}	74^{a}	41^{a}	--
October	189^{a}	155^{a}	193^{a}	222^{a}	210^{a}	--
November	337^{a}	189^{b}	59^{b}	81^{b}	57^{b}	--
December	460^{a}	207^{b}	74^{c}	100^{c}	15^{c}	--
1983						
March	651^{a}	318^{b}	63^{c}	100^{c}	67^{c}	--
May	569^{a}	376^{a}	144^{b}	156^{b}	129^{b}	--
June	483^{a}	303^{ab}	154^{b}	150^{b}	158^{b}	--
July	326^{a}	93^{b}	--[+]	1^{b}	1^{b}	0^{b}
August	222^{a}	140^{b}	--	7^{c}	15^{c}	4^{c}
September	181^{a}	52^{b}	--	11^{c}	30^{c}	4^{c}
October	348^{a}	267^{b}	--	89^{c}	96^{c}	4^{c}
1984						
April	433^{a}	381^{a}	--	119^{b}	76^{b}	14^{b}
June	586^{a}	314^{b}	--	65^{c}	37^{c}	14^{c}
July	549^{a}	267^{b}	--	123^{c}	108^{c}	0^{c}

[*] Densities with a common letter as a superscript in a horizontal row are not significantly different for the noted month ($P<0.05$).

[+] In June 1983, the low application plots became the very high application plots.

[**] Background sample taken before any wastes were applied or the plots were rototilled.

TABLE I-2
AVERAGE EARTHWORM BIOMASS FOUND IN THE FIELD PLOTS (g/m^2)

	Type of Application					
Date	Natural Controls	Rototilled Controls	Low	Medium	High	Very High
1982						
May**	60^{a}*	80^{a}	79^{a}	98^{a}	90^{a}	--+
June	73^{a}	43^{a}	32^{a}	39^{a}	31^{a}	--
July	34^{a}	24^{ab}	5^{c}	6^{c}	17^{bc}	--
August	31^{a}	14^{a}	8^{a}	11^{a}	21^{a}	--
September	15^{a}	13^{a}	5^{a}	8^{a}	6^{a}	--
October	85^{a}	61^{a}	48^{a}	49^{a}	41^{a}	--
November	125^{a}	50^{b}	17^{c}	15^{c}	4^{c}	--
December	146^{a}	46^{b}	12^{c}	17^{c}	4^{c}	--
1983						
March	127^{a}	74^{b}	5^{c}	18^{c}	11^{c}	--
May	174^{a}	136^{a}	25^{b}	30^{b}	26^{b}	--
June	101^{a}	96^{a}	25^{b}	25^{b}	30^{b}	--
July	54^{a}	20^{b}	--+	2^{c}	2^{c}	0^{c}
August	24^{a}	23^{a}	--	1^{b}	1^{b}	6^{b}
September	25^{a}	8^{b}	--	1^{b}	3^{b}	2^{b}
October	93^{a}	68^{b}	--	11^{c}	13^{c}	1^{c}
1984						
April	119^{a}	151^{a}	--	41^{b}	19^{b}	2^{b}
June	122^{a}	104^{a}	--	29^{b}	13^{b}	3^{b}
July	119^{a}	44^{b}	--	37^{b}	28^{b}	0^{b}

*Numbers with a common letter as a superscript in a row are not significantly different for the noted month ($P<0.05$).

+In June 1983, the low application plots became the very high application plots.

**Background sample taken before any wastes were applied or the plots were rototilled.

TABLE J-1
METAL CONCENTRATIONS[+] IN EARTHWORMS OF THE NATURAL CONTROL PLOTS
(mg/kg MOISTURE FREE EARTHWORM TISSUE)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum	7,900	6,400	8,400	6,800	9,100	10,100	8,100
(Al)	±1,100	±1,800	±3,200	±920	±1,400	±1,200	±1,300
Cadmium	4	4	4	3	4	4	3
(Cd)	±1	±1	±1	±1	±1	±1	±0
Calcium	3,500	3,500	4,200	3,700	3,700	3,600	3,700
(Ca)	±800	±650	±750	±540	±600	±200	±600
Chromium	10	8	10	8	11	13	10
(Cr)	±1	±3	±4	±1	±2	±2	±2
Copper	12	11	12	12	12	10	11
(Cu)	±3	±1	±2	±2	±2	±1	±1
Iron	9,600	7,900	10,500	10,200	9,300	11,400	9,700
(Fe)	±1,500	±1,900	±3,700	±1,000	±1,300	±1,300	±1,100
Lead	12	9	13	15	9	9	7
(Pb)	±2	±2	±4	±2	±2	±1	±1
Magnesium	2,200	2,000	2,200	2,100	2,200	2,400	2,000
(Mg)	±200	±300	±550	±150	±200	±200	±200
Manganese	180	110	240	180	170	210	180
(Mn)	±39	±30	±120	±63	±40	±40	±42
Nickel	11	8	10	11	16	12	9
(Ni)	±1	±3	±3	±2	±5	±2	±1
Potassium	6,900	7,500	6,100	7,200	6,400	5,700	6,000
(K)	±740	±1,400	±900	±640	±2,200	±300	±500
Sodium	2,300	2,800	2,300	2,700	2,600	2,400	2,300
(Na)	±370	±390	±210	±150	±300	±400	±200
Titanium	48	36	23	24	68	37	45
(Ti)	±3	±17	±6	±2	±15	±13	±15
Vanadium	13	10	12	9	15	16	13
(V)	±2	±3	±4	±1	±2	±2	±2
Zinc	220	260	220	240	290	270	260
(Zn)	±51	±66	±59	±47	±70	±50	±38

+ Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

TABLE J-2
METAL CONCENTRATIONS[+] IN EARTHWORMS OF THE ROTOTILLED CONTROL PLOTS
(mg/kg MOISTURE FREE EARTHWORM TISSUE)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum	6,300	3,700	11,600	6,300	12,100	10,000	8,400
(Al)	±650	±320	±2,900	±1,900	±1,400	±1,600	±1,100
Cadmium	5	5	3	5	4	4	3
(Cd)	±2	±1	±1	±0	±1	±1	±0
Calcium	3,300	2,600	3,500	6,000	3,600	3,800	3,900
(Ca)	±180	±810	±500	±2,990	±410	±700	±400
Chromium	7	15	13	9	16	12	12
(Cr)	±1	±18	±2	±1	±3	±2	±3
Copper	12	29	13	11	12	11	10
(Cu)	±1	±3	±3	±0	±2	±3	±1
Iron	7,800	5,900	13,700	8,400	12,200	11,500	10,300
(Fe)	±71	±1,000	±1,500	±2,000	±700	±2,000	±1,400
Lead	11	30	15	15	11	9	8
(Pb)	±2	±4	±2	±7	±2	±1	±1
Magnesium	2,000	1,900	2,700	2,100	2,600	2,400	2,100
(Mg)	±43	±680	±240	±270	±200	±300	±200
Manganese	150	110	240	190	240	213	210
(Mn)	±45	±20	±81	±76	±66	±28	±63
Nickel	10	6	13	10	17	27	10
(Ni)	±3	±1	±1	±1	±5	±7	±2
Potassium	7,300	7,100	5,500	6,200	7,000	5,500	6,000
(K)	±1,300	±1,200	±1,200	±1,500	±240	±1,400	±700
Sodium	2,700	3,200	1,900	2,600	2,500	2,700	2,300
(Na)	±320	±410	±370	±690	±260	±700	±400
Titanium	39	45	34	27	84	44	37
(Ti)	±11	±13	±5	±4	±13	±15	±8
Vanadium	10	8	16	10	19	16	13
(V)	±1	±3	±3	±1	±3	±3	±2
Zinc	310	280	180	350	250	220	230
(Zn)	±70	±47	±12	±20	±33	±42	±22

[+] Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

TABLE J-3
METAL CONCENTRATIONS[+] IN EARTHWORMS OF THE LOW APPLICATION PLOTS
(mg/kg MOISTURE FREE EARTHWORM TISSUE)

Metal	Sampling Date				
	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83
Aluminum (Al)	5,000 ±880	5,400 ±2,500	8,700 ±1,600	5,900 ±1,220	9,400 ±2,100
Cadmium (Cd)	3 ±1	6 ±2	3 ±2	7 ±2	9 ±5
Calcium (Ca)	4,400 ±1,600	2,600 ±410	5,100 ±2,400	4,000 ±860	3,600 ±600
Chromium (Cr)	6 ±1	14 ±8	11 ±3	8 ±0	24 ±11
Copper (Cu)	11 ±3	20 ±11	13 ±2	17 ±6	26 ±12
Iron (Fe)	6,200 ±620	6,500 ±3,300	11,100 ±2,400	8,000 ±1,400	8,800 ±1,400
Lead (Pb)	10 ±5	23 ±20	16 ±2	15 ±0	25 ±13
Magnesium (Mg)	1,800 ±180	1,700 ±280	2,300 ±330	1,900 ±190	2,200 ±200
Manganese (Mn)	150 ±24	110 ±50	210 ±55	150 ±24	170 ±46
Nickel (Ni)	9 ±4	14 ±8	11 ±2	12 ±1	18 ±6
Potassium (K)	8,400 ±870	7,300 ±1,900	6,300 ±980	8,000 ±600	6,500 ±2,200
Sodium (Na)	2,900 ±190	2,700 ±170	2,400 ±470	3,900 ±290	3,100 ±600
Titanium (Ti)	40 ±15	102 ±10	22 ±1	-** -	140 ±60
Vanadium (V)	8 ±1	16 ±7	12 ±2	8 ±0	18 ±4
Zinc (Zn)	260 ±32	320 ±90	250 ±38	230 ±70	300 ±49

[+] Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

**Less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE J-4
METAL CONCENTRATIONS[+] IN THE EARTHWORMS OF THE MEDIUM APPLICATION PLOTS
(mg/kg MOISTURE FREE EARTHWORM TISSUE)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum	5,900	5,900	8,900	7,600	9,200	11,000	9,500
(Al)	±770	±2,300	±1,800	±3,400	±1,800	±3,000	±1,100
Cadmium	4	5	4	4	6	5	3
(Cd)	±2	±1	±1	±1	±3	±2	±1
Calcium	3,700	3,200	4,700	3,100	3,800	4,400	5,100
(Ca)	±960	±300	±1,800	±560	±500	±700	±700
Chromium	7	11	13	11	18	20	19
(Cr)	±1	±4	±2	±2	±14	±0	±5
Copper	10	16	36	12	17	12	12
(Cu)	±1	±4	±4	±1	±6	±0	±1
Iron	7,400	7,400	10,800	8,700	9,100	11,600	10,700
(Fe)	±1,200	±2,300	±1,800	±4,100	±1,500	±3,100	±1,200
Lead	8	19	16	12	19	----**	10
(Pb)	±1	±11	±6	±0	±9	----	±1
Magnesium	1,800	1,900	2,300	1,900	2,200	2,600	2,200
(Mg)	±190	±290	±130	±450	±100	±500	±100
Manganese	140	140	210	180	150	178	205
(Mn)	±32	±52	±51	±38	±46	±70	±60
Nickel	8	11	12	12	18	17	10
(Ni)	±2	±4	±3	±2	±5	±0	±2
Potassium	7,300	8,200	6,400	6,900	6,500	6,500	6,100
(K)	±580	±1,100	±1,600	±2,200	±600	±800	±500
Sodium	2,600	3,500	2,700	3,200	2,800	2,900	2,400
(Na)	±370	±360	±640	±1,600	±300	±400	±200
Titanium	32	79	29	28	100	80	57
(Ti)	±10	±51	±9	±0	±41	±0	±15
Vanadium	9	12	12	12	16	23	14
(V)	±1	±3	±3	±3	±1	±0	±3
Zinc	230	240	240	430	230	220	390
(Zn)	±32	±25	±58	±320	±21	±8	±120

[+] Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

** Less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE J-5
METAL CONCENTRATIONS[+] IN THE EARTHWORMS OF THE HIGH APPLICATION PLOTS
(mg/kg MOISTURE FREE EARTHWORM TISSUE)

	Sampling Date						
Metal	6/4/82*	6/21/82	10/5/82	11/9/82	6/6/83	10/25/83	6/6/84
Aluminum	6,800	3,500	11,400	8,300	1,300	9,000	8,700
(Al)	±1,500	±1,400	±1,500	±0	±2,500	±4,100	±1,500
Cadmium	4	5	3	-**	4	6	4
(Cd)	±1	±3	±1	-	±1	±3	±1
Calcium	4,300	2,900	4,300	7,000	3,400	4,500	5,100
(Ca)	±1,400	±440	±1,500	±0	±500	±900	±900
Chromium	8	7	19	-	21	19	21
(Cr)	±2	±4	±7	-	±8	±3	±7
Copper	12	12	16	-	12	15	14
(Cu)	±1	±2	±4	-	±1	±3	±2
Iron	5,900	7,800	13,800	±14,000	±12,500	10,100	9,700
(Fe)	±2,700	±3,100	±500	±0	±1,500	±4,400	±1,700
Lead	8	11	21	-	15	15	10
(Pb)	±2	±3	±15	-	±6	±0	±2
Magnesium	2,100	1,800	2,600	2,700	2,600	2,200	2,100
(Mg)	±270	±470	±96	±0	±300	±600	±200
Manganese	240	160	300	300	250	280	210
(Mn)	±100	±70	±64	±0	±72	±150	±27
Nickel	9	8	16	-	20	16	11
(Ni)	±2	±3	±6	-	±9	±1	±3
Potassium	7,300	7,100	5,700	6,200	6,800	6,400	5,900
(K)	±860	±520	±1,100	±0	±600	±700	±600
Sodium	2,600	3,200	2,600	2,700	2,900	3,200	2,500
(Na)	±230	±270	±350	±0	±200	±600	±200
Titanium	39	34	40	-	120	80	57
(Ti)	±10	±20	±6	-	±28	±8	±15
Vanadium	11	9	16	-	20	19	14
(V)	±3	±5	±2	-	±6	±2	±3
Zinc	230	250	220	240	200	200	390
(Zn)	±30	±30	±45	±0	±60	±65	±100

[+] Average and standard deviation of the data from the four plots on the noted sampling data.

* Background sample taken before any wastes were applied or the plots were rototilled.

** Less than the noted detection limit of the analytical methods when calculated in terms of these units.

TABLE J-6
METAL CONCENTRATIONS[+] IN EARTHWORMS FROM THE VERY HIGH APPLICATION PLOTS (mg/kg MOISTURE FREE EARTHWORM TISSUE)

	Sampling Date	
Metal	6/6/83	6/6/84
Aluminum (Al)	9,400 ±2,100	3,500 ±1,200
Cadmium (Cd)	4 ±1	4 ±0
Calcium (Ca)	3,600 ±400	6,300 ±1,300
Chromium (Cr)	16 ±3	12 ±3
Copper (Cu)	12 ±2	10 ±1
Iron (Fe)	12,200 ±700	10,300 ±1,400
Lead (Pb)	11 ±2	8 ±1
Magnesium (Mg)	2,600 ±200	2,100 ±200
Manganese (Mn)	240 ±66	210 ±63
Nickel (Ni)	17 ±5	10 ±2
Potassium (K)	7,000 ±200	6,000 ±700
Sodium (Na)	2,500 ±300	2,300 ±400
Titanium (Ti)	84 ±13	37 ±8
Vanadium (V)	19 ±3	13 ±2
Zinc (Zn)	250 ±33	232 ±22

[+] Average and standard deviation of the data from the four plots on the noted sampling data.

TABLE K-1
QUALITY ASSURANCE ANALYSES OF THE STANDARD SLUDGE SUPPLIED BY THE PROJECT OFFICER - OIL AND GREASE RESULTS

Date of Analysis	Average Oil and Grease* (g/kg MFS)	Date of Analysis	Average Oil and Grease* (g/kg MFS)
1983		1984	
1/9	44.98	1/26	45.98
2/9	44.10	3/1	50.07
2/10	44.42	6/3	48.14
2/14	44.10	6/7	51.14
3/25	43.50	7/2	48.55
6/14	49.16		
7/3	47.62		
8/1	51.22		
8/15	52.60		
8/19	52.88		
11/2	49.28		
12/2	45.00		

*Two samples of the standard sludge were analyzed on each date.

Note: 95% of these data were within one standard deviation of the mean and all of the data were within two standard deviations of the mean.

The average oil and grease concentration and 95% confidence limits for the standard sludge, as supplied by EPA, were: average = 52.68 g/kg MFS, 95% limits = 26.1 to 79.3 g/kg MFS.

The results obtained from these quality assurance evaluations were: average = 47.7 g/kg MFS, 95% limits = 46.3 to 49.2 g/kg MFS.